APPENDIX A
LABORATORY STANDARD OPERATING PROCEDURES

QAM CHANGE FORM

SOP Title: Quality Assurance Manual
SOP Code: HE-QAM
SOP Revision No.: 14
SOP Date: 11/10/2014
SOP Section(s) Affected by Change: Figure 11.1,11.2, Appendix B

Description of Change: 1.Replace Columbia Analytical Service CoC with an ALS HRMS COC 2.Replace Columbia Analytical Service SR with an ALS HRMS SR 3.Remove Jim Plassard as the Lab Director and add Hoai Van. Remove Rebecca Pierrot as the QA/EHS Manager and add Tonia King-Cormier. Remove Jim Plassard's resume and add Hoai Van's resume. Remove Rebecca Pierrot's resume and add Tonia King-Cormier's resume. Remove Jim Plassard and Rebecca Pierrot as approved signatories for final analytical reports and add Hoai Van's and Tonia King-Cormier

Reason(s) for Change(s): Change of company name and address as well as change of management

Change(s) Submitted byTonia King-Cormier	Date: 4/2/2015

Approvals:

Technical Reviewer Signature:	Date: 04/02/2015
QA PM Signature: Tonia King-Cormier	Date: 4/2/2015
Department Supervisor/Manager Signature:	Date: 4/2/15

Change(s) Effective Date: 4/2/2015

ALS Environmental

QUALITY ASSURANCE MANUAL

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QUALITY ASSURANCE MANUAL

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Approved By: _____ Date: 11-05-14
Laboratory Director – Jim Plassard

Approved By: _____ Date: 11.5.14
QA Manager – Rebecca Pierrot

Approved By: _____ Date: 11/05/14
Technical Director – Lan Le

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1) Introduction and Scope

ALS Environmental, Houston HRMS is a professional analytical services laboratory, which performs chemical analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in SOP CE-GEN001, *Laboratory Ethics and Data Integrity* and in this Quality Assurance Manual (QAM). ALS – Houston HRMS is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented, and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs, such as the Department of Defense (DOD) Environmental Laboratory Accreditation Program, as well as client's quality objectives. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. The laboratory maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

This QAM is applicable to the facility listed on the title page. The information in this manual has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025:2005, and the U.S. Department of Defense Quality Systems Manual for Environmental Laboratories, 2013. A glossary of pertinent terms and acronyms is included in Appendix A.

2) Organization

The ALS Environmental, Houston HRMS staff, consisting of approximately 16 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks. All employees share the responsibility for maintaining and improving the quality of our analytical services.

ALS – Houston HRMS is legally identifiable as ALS Group USA, Corp., dba ALS Environmental. ALS Group USA, Corp. is a component of ALS Limited, a publicly held Australian company. The ALS Global website may be referred to for corporate ownership information (www.Alsglobal.com/Our-Company). The laboratory is divided into operational and managerial units based upon specific disciplines. Each department is responsible for establishing, maintaining, and documenting QA and QC practices meeting laboratory needs. An organizational chart of the laboratory, as well as resumes of key personnel can be found in Appendix B. This laboratory organization is designed so that potential conflict of interest is avoided, and such that an adequate amount of supervisory personnel are in place to provide oversight and supervision of day to day operations.

3) Management

The purpose of the QA program at ALS Environmental, Houston HRMS, is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement:

"The mission of ALS Environmental, Houston HRMS is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development, and company commitment."

3.1 Quality Management Systems

In support of this mission, the laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Manager (QA Manager) with corporate oversight by the Chief Quality Officer (CQO). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (NELAC 2003, 2009 and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions, and documentation are defined in both administrative and technical SOPs. Figure 3-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

- Accreditation and certification program compliance
- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Document Control
- Demonstration of Capability
- Analytical Traceability
- Ethics training and data integrity processes
- Corrective action procedures
- Statistical Control Charting
- Management Reviews

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits
- Periodic reports to management
- Analysis of customer feedback
- Proficiency testing

The responsibilities of key positions within the laboratory are described below. Table 3-1 lists the ALS –Houston HRMS personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. In the event that work is stopped in response to quality problems, as described below, only the Laboratory Director, or Quality Assurance Manager has the authority to resume work.

Laboratory Director - The role of the Laboratory Director is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the (Location) facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

Technical Director – The role of the Technical Director is to oversee the day-to-day technical production of analytical data. This oversight is achieved through the following activities: monitoring quality control performance, monitoring the validity of generated data to ensure reliable data, corroborating the analysis performed, and certifying demonstrations of capability. The Technical Director certifies that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited; reviews new methods for their applicability to a project, implements new methodology at the facility, and directs, trains and supervises individuals participating in this effort; in the case of the Technical Director absence, the Laboratory Operations Director or a Technical Supervisor shall maintain these duties. Requires a BS or BA degree in Science, Engineering or Management (with at least 24 college semester credits in chemistry), and five years technical supervisory experience in environmental laboratory operations. This individual is an approved signatory for all facility policies and procedures, as well as training documentation.

Quality Assurance Manager - The Quality Assurance Manager (QA Manager) has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that personnel understand the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all be used to support quality system implementation. The QA Manager is

responsible for ensuring compliance with NELAC standards (and ISO, DoD QSM, etc. as applicable). The QA Manager works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA Manager is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting proficiency testing studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; and performing internal QA audits.

The QA Manager reports directly to the Laboratory Director and reports indirectly to the ALS Manager of Quality Assurance, USA. It is important to note that when evaluating data, the QA Manager does so in an objective manner and free of outside, or managerial, influence.

The Manager of Quality Assurance, USA is responsible for the overall QA program at all the ALS Environmental laboratories. The Manager of Quality Assurance, USA is responsible for oversight of QA Managers regulatory compliance efforts (NELAC, ISO, DOD, etc). And may perform internal audits to evaluate compliance. The Manager of Quality Assurance, USA approves company-wide SOPs and provides assistance to the laboratory QA staff and laboratory managers as necessary.

Deputy Laboratory Director and QA Manager - In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Technical Director (for the Laboratory Director) and the Laboratory Director (for the QA Manager).

Environmental Health and Safety (EH&S) Officer – The Environmental Health and Safety Officer (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to ALS North America EH&S Manager.

Client Services Manager (CSM) – The CSM is responsible for all aspects of client services within the laboratory. This includes management and oversight of Project Managers, electronic deliverables, and support functions. The laboratory provides a complete interface with clients from initial project specification to final deliverables. The Client Services Manager has the responsibility and authority to stop work in response to accreditation/certification or quality problems, or in response to similar subcontractor quality problems. The ALS – Houston HRMS Laboratory Director serves in this capacity.

Department Managers and Supervisors – Each manager or supervisor has the responsibility to ensure that QA and QC functions are carried out as specified when executing analyses and related tasks to ensure the production of high quality data. Managers and bench-level supervisors monitor the day-to-day operations to ensure that productivity and data quality objectives are met. A department manager has the

authority to stop work in response to quality problems in their area. Managers and supervisors are responsible for ensuring that analysts perform testing according to applied methods, SOPs, and QC guidelines particular to the laboratory department.

Sample Management Office (SMO) – The Sample Management Office plays a key role in the laboratory QA program by handling all activities associated with receiving, storage, and disposal of samples, and maintaining documentation for all samples received. SMO staff is also responsible for the proper disposal of samples after analysis. The Laboratory Director oversees SMO and bottle preparation functions.

Information Technology (IT) – IT staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) support, and data back-up, archival, and integrity operations.

3.2 Ethics, Professional Conduct and Data Integrity

One of the most important aspects of the success of ALS – Houston HRMS is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within ALS – Houston HRMS as well as established laboratory practices. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

All employees are required to sign and adhere to the requirements set forth in the *ALS Code of Conduct Policy* and agree to the *Confidentiality Agreement* (Appendix C).

3.2.1 Professional Conduct

To promote quality, ALS – Houston HRMS requires certain standards of conduct and ethical performance among employees. The following examples of documented ALS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.

3.2.2 Confidentiality

It is the responsibility of all laboratory employees to safeguard sensitive company information, client data, records, and information; and matters of

national security concern should they arise. The nature of our business and the well-being of our company and of our clients is dependent upon protecting and maintaining confidential and/or proprietary company and client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential.

Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

3.2.3 Prevention and Detection of Improper, Unethical, or Illegal Actions

It is the intention of the laboratory to proactively prevent and/or detect any improper, unethical, or illegal action conducted within the laboratory. This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel in their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review, and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements for internal QA audits are described in SOP CE-QA001, Internal Audits. All aspects of this program are documented and retained on file according to the company policy on record retention.

The ALS Employee Handbook also contains information on the ALS ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

3.2.4 Laboratory Data Integrity and Ethics Training

Each employee receives in-depth "core" Data Integrity/Ethics Training. New employees are given a QA and Ethics orientation within the first month of hire, followed by the core training within 1 year of hire. On an ongoing basis, all employees receive semi-annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA Manager to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

3.2.5 Management and Employee Commitment

The laboratory makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the the laboratory Employee Handbook. This includes:

- ALS Open Door Policy (ALS Employee Handbook) – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.
- FairCall – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (ALS Employee Handbook) – To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

Table 3-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jim Plassard, B.S.	21	Laboratory Director
Lan Le, Ph.D.	26	Technical Director, Analytical Supervisor
Rebecca Pierrot, B.S.	13	Quality Assurance Manager, Environmental Health and Safety Officer
Nicole Brown, B.S.	7	Project Manager
Arthi Kodur, M.S.	7	Project Manager
Mike Sullivan, B.S.	14	Information Technology
Les Arnold	31	Director of Operations, Eastern USA

4) Document Control

Procedures for control and maintenance of documents are described in SOP CE-GEN005, *Document Control.* The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled laboratory documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QA Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following the SOP CE-QA007, *Making Entries onto Analytical Records*. Entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in SOP HE-QA002, *Report Data and Record Archiving.*.

External documents relative to the management system are managed by the QA Manager. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a

master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

5) Review of Requests, Tenders and Contracts

Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved. Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work. If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

6) Subcontracting of Tests

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another ALS laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described SOP CE-QA004, *Qualification of Subcontract Laboratories*. The Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

7) Purchasing Services and Supplies

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. CE-QA012*, Quality of Reagents and Standards,* and HE-EXT006, *Procedures for Standard Preparation,* provide default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in SOP CE-GEN007, *Procurement and Control of Laboratory Services and* Supplies.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for

unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Checking New Lots of Chemicals for Contamination* (ADM-CTMN).

8) Service to the Client

The laboratory utilizes a number of processes to ensure that adequate resources exist to meet service demands. Senior staff meetings, tracking of outstanding proposals and a current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. Status/production meetings are conducted regularly with the laboratory and Project Managers to inform the staff of the status of incoming work, future projects, and project requirements.

The Project Manager is a scientist assigned to each client to act as a technical liason between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project and contract requirements. This entails coordinating with the laboratory staff to ensure that client-specific needs are understood and that the services provided are properly executed and satisfy the requirements of the client.

Laboratory management also monitors a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. This includes on-time performance, customer complaints, training reports and non-conformity reports. A frequent assessment is made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

All Requests for Proposal (RFP) documents are reviewed by the Project Manager and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that potentially cannot be met are noted and communicated to the client, as well as requesting the client to provide any applicable project specific Quality Assurance Project Plans (QAPPs).

When a client requests a modification to an SOP, policy or standard specification, the Project Manager will discuss the proposed deviation with the Laboratory Manager, and department supervisors to obtain approval for the deviation. The QA Manager may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A project-specific communication form, or similar, may be used to document such deviations.

The laboratory affords clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The SOP CE-GEN010, *Handling Customer Feedback* is in place for these events.

9) Complaints

In addition to project communication and internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The procedure is described in SOP CE-GEN010, *Handling Customer Feedback*. The person who initially receives feedback in the form of a complaint (typically the Project Manager) is responsible for documenting the

complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented

10) Facilities and Equipment

The laboratory features 8,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each workspace. Also, the laboratory minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Semi-volatile Organics Sample Preparation Laboratory
- High Resolution Gas Chromatography/High Resolution Mass Spectrometry Laboratory
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation, and shipping provide for the efficient and safe handling of a variety of sample types. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Refer to Appendix D for a Laboratory Floor Plan and Appendix E for a list of major equipment, illustrating the laboratory's overall capabilities and depth.

11) Sample Management

11.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. The laboratory recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

1. Amount of sample taken
2. Type of container used
3. Type of sample preservation
4. Sample storage time
5. Proper custodial documentation

The laboratory uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 23 for complete citations). The container, preservation and holding time information for these references is summarized in Table 11-1 for soil, water, and drinking water. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

The laboratory routinely provides sample containers for our clients. Containers are purchased as pre-cleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Our sample kits typically consist of foam-lined, pre-cleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 11-1 shows the chain-of-custody form routinely used by the laboratory and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to the laboratory

If any returning shipping cooler exhibits an odor or other abnormality upon receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. The laboratory keeps client-specific shipping requirements on file and utilizes major transportation carriers necessary to meet sample-shipping requirements (same-day, overnight, etc.)

When the laboratory ships environmental samples to other laboratories for analysis, similar sample integrity processes are used to ensure preservation and proper handling, and to avoid any possible breakage, cross-contamination of samples, or identification of problems. Alternatively, the receiving laboratory's procedures may be specified. Chain of custody is maintained during the process.

11.2 Sample Receipt and Handling

Standard procedures are established for the receiving of samples into the laboratory and are found in SOP HE-SMO001, *Sample Receiving*. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

When samples are received by the laboratory, a Cooler Receipt and Preservation Check Form (CRF - Figure 11-2) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Verification of sample integrity includes the following activities:

6. Assessment of custody seal presence/absence, location and signature;
7. Temperature of sample containers upon receipt;
8. Chain of custody documents properly used (entries in ink, signature present, etc.);
9. Sample containers checked for integrity (broken, leaking, etc.);
10. Sample is clearly marked and dated (bottle labels complete with required information);
11. Appropriate containers (size, type) are received for the requested analyses;
12. The minimum amount of sample material is provided for the analysis;
13. Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.); and
14. Assessment of proper sample preservation (if inadequate, corrective action is employed)

Samples are logged into a Laboratory Information Management System (LIMS). Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Manager and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample container is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Manager for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. The laboratory one large walk-in cold storage units which house the majority of sample containers received at the laboratory. the laboratory also has three sub-zero freezers capable of storing samples at -20° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily, which is recorded on the daily temperature log for each unit.

The laboratory adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is

printed or written on the analytical raw data. Table 11-1 summarizes the typical holding times for analyses performed by the laboratory.

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous, soil, and tissue samples are retained for 30 days. Extracts are stored for up to one year. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *ALS Environmental Health and Safety Manual*, and in the SOP HE-SMO003, *Waste Disposal*. All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

11.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. Photographic records of each sample are captured at sample receipt. This is described in the SOP HE-SMO001, *Sample Receipt*. Figure 11-1 is a copy of the chain-of-custody form routinely used at the laboratory. Figure 11-2 is a copy of the cooler receipt form routinely used at the laboratory.

Facility security and access is important in maintaining the integrity of samples received at the laboratory. Access to the laboratory facility is limited by use locked exterior doors with a coded/card entry. In addition, the sample storage area within the laboratory is a controlled access area, limited to ALS – Houston HRMS personnel and is locked after hours. The laboratory facility is equipped with an alarm system.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts the analyst documents custody of the sample extract by signing on the custody record, that they have accepted custody. Sample tracking procedures are outlined in SOP HE-SMO001, *Sample Receipt.*

11.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. LIMS codes are chosen at the time of project set up to identify the analytical method used for analysis. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Manager ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific

requirements, a Tier V form (Figure 7-3) is used and accompanies the service request form.

Table 11-1

Sample Preservation and Holding Times

DETERMINATION	MATRIX[a, b]	CONTAINER[c]	PRESERVATION[d]	MAXIMUM HOLDING TIME[e]
Dioxins/Furans by EPA 8290	W	G	4°C	30 days
Dioxins/Furans by EPA 8280A	W	G	4°C	30 days
Dioxins/Furans by EPA 8290	S	G	4°C	30 days, 1 year if frozen
Dioxins/Furans by EPA 8280A	S	G	4°C	30 days, 1 year if frozen
Dioxins/Furans by EPA 1613B	W, DW	G	0-4°C	1 year
Dioxins/Furans by EPA 1613B	S	G	-20°C to -10°C	1 year
Dioxins/Furans by EPA 23	A	XAD	0-6°C	30 days
Dioxins/Furans by EPA 1613B	T	G	-20°C to -10°C	1 year
Dioxins/Furans by EPA 8290	T	G	-20°C to -10°C	30 days
Dioxins/Furans by EPA TO-9A	A	PUF	<4°C	7 days
Polychlorinated Biphenyls (PCBs) by EPA 1668	W	G	<6°C	1 year
Polychlorinated Biphenyls (PCBs) by EPA 1668	S	G	-20°C to -10°C	1 year
Polychlorinated Biphenyls (PCBs) by EPA 1668	T	G	-20°C to -10°C	1 year
Polychlorinated Biphenyls (PCBs) by CARB[f] 428	A	XAD	Ambient	45 days
Polycyclic Aromatic Hydrocarbons (PAHs) by CARB[f] 429	A	XAD	<4°C	21 days
Pesticides by EPA 1699	W	G	<6°C	7 days
Pesticides by EPA 1699	S	G	-20°C to -10°C	1 year

a A = Air, DW = Drinking Water, S = Soil or Sediment, T = Tissue, W = Water
b Amount of sample required: DW, W = 2x1L; S, T = 50g; A = contents of 1 trap
c G = Glass, PUF = Polyurethane foam plug, XAD = XAD filled glass trap
d NELAC §5.5.8.4(a)(1) – For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.
e Holding times listed for samples only. Extract holding times can be found in referenced technical methods.
f CARB = California Air Resource Board

Figure 11-1
Chain of Custody Form

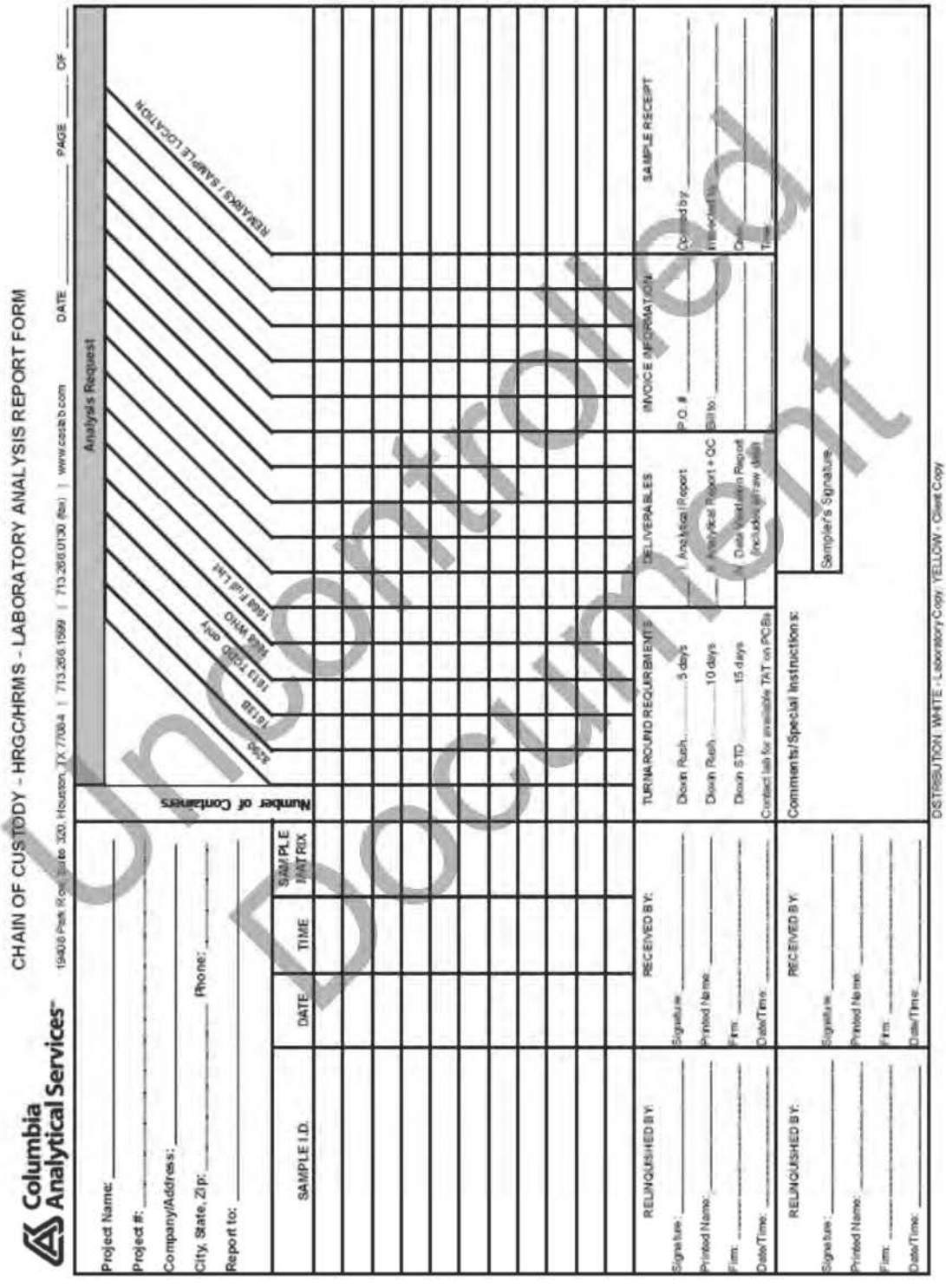


Figure 11-2
Cooler Reciept and Preservation Form

Columbia Analytical Services

Cooler Receipt Form

Project Chemist

Client/Project | Service Request

Date Received: | Technician | Date Logged In: | Technician

1. Method of delivery: ○ US Mail ○ Fed Ex ○ UPS ○ DHL ○ Courier ○ Client
2. Samples received in: ○ Cooler ○ Box ○ Envelope ○ Other
3. Were custody seals on coolers? ○ Yes ○ No ○ N/A — If yes, how many and where?
   Were they intact? ○ Yes ○ No ○ N/A
   Were they signed and dated? ○ Yes ○ No ○ N/A
4. Method of delivery: ○ Inserts ○ Baggies ○ Bubble Wrap ○ Gel Packs ○ Wet Ice ○ Sleeves ○ Other
5. Foreign or Regulated Soil? ○ Yes ○ No — Location of Sampling:

Cooler Tracking Number	COC ID	Date Opened	Time Opened	Opened By	Temp. °C	Temp Blank?	Filed
						☐	☐
						☐	☐
						☐	☐
						☐	☐
						☐	☐

5. Were custody papers properly filled out (ink, signed, dated, etc)? ○ Yes ○ No ○ N/A
6. Did all bottles arrive in good condition (not broken, no signs of leakage)? ○ Yes ○ No ○ N/A
7. Were all sample labels complete (i.e., sample ID, analysis, preservation, etc)? ○ Yes ○ No ○ N/A
8. Were appropriate bottles/containers and volumes received for the requested tests? ○ Yes ○ No ○ N/A
9. Did sample labels and tags agree with custody documents? ○ Yes ○ No ○ N/A

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count	Bottle Type	Out of Temp	Broken	Date	Technician
			☐	☐		
			☐	☐		
			☐	☐		
			☐	☐		

Notes, Discrepancies, & Resolutions:

Print Form | Reset Form

12) Analytical Procedures

The laboratory employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, EPA 40CFR parts 136 and 141, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 23. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection/reporting limit, the expected concentration of the analyte being measured, method selectivity, accuracy, and precision, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by the laboratory is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix J.

12.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

The laboratory maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements as described in SOP CE-GEN009, *Preparation of Standard Operating Procedures*. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The procedures for document control are described in SOP CE-GEN005, *Document Control,* In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in SOP CE-QA007, *Making Entries into Logbooks and onto Benchsheets.* Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval. A list of current SOPs is given in Appendix G

12.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the Project Manager handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the Laboratory Director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

12.3 Modified Procedures

The laboratory strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

12.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that the laboratory has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1. The number of (field) samples in a batch is not to exceed 20.
2. All (field) samples in a batch are of the same matrix.
3. The QC samples to be processed with the (field) samples include:
   - Method Blank (a.k.a. Laboratory Reagent Blank)
   - Laboratory Control Sample
   - Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*
   - Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4. A single lot of reagents is used to process the batch of samples.
5. Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.
6. Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.
7. Field samples are assigned to batches commencing at the time that sample processing begins. For analysis of organic constituents, it begins when the samples are extracted.
8. The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple

instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

9. The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
10. Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
11. Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

12.5 Specialized Procedures

The laboratory not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications.

12.6 Sample Cleanup

The laboratory commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

13) Measurement Traceability and Calibration

All equipment and instruments used at the laboratory are operated, maintained, and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at the laboratory are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified as outlined in SOP HE-QA005, *Calibration Verification of Support Equipment*. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment shown by verification to be malfunctioning or defective is taken out of service until it is repaired. When an instrument is taken out of service, an "Out of Service" sign is placed on the instrument. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

13.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, ovens and standards refrigerators. Temperature monitoring logs are maintained, which contain daily-recorded temperatures, identification, and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the SOP HE-QA005, *Calibration Verification of Support Equipment*.

The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked quarterly against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an as described in SOP HE-QA005, *Calibration Verification of Support Equipment*.

13.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP HE-QA005, *Calibration Verification of Support Equipment*.

The weights are recertified using NIST traceable standards by an accredited metrology organization as described by SOP HE-QA005, *Calibration Verification of Support Equipment.*

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on an annual basis, by an accredited metrology organization, or when the balance shows signs of degradation.

13.3 Water Purification Systems

The laboratory uses a water purification system designed to produce deionized water meeting method specifications. The system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water.

13.4 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors

have fulfilled the requirements for ISO 9001 certification and/or are accredited by A2LA. the laboratory relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Cambridge Isotope Laboratories (CIL), Wellington Laboratories, and Accustandard are examples of the vendors used. Reference material information is recorded in the "Materials Logbook" in LIMS and materials are stored under conditions that provide maximum protection against deterioration and contamination. Entries in the Materials Logbook include such information as an assigned LIMS identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the SOP HE-EXT006, *Preparation of Standard Solutions* and are entered in to LIMS for tracking purposes. Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material.

13.5 High Resolution GC/MS Systems

All HRGC/HRMS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using perfluorokerosene (PFK). Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed.

13.6 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (SMO-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks. Pipettes are serviced and calibrated annually by an approved vendor.

14) Assuring the Quality of Results

A primary focus of the laboratory's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. the laboratory has established Quality Control (QC) objectives for precision and accuracy that are used to

determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

14.1 Quality Control Objectives

14.1.1 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

14.1.2 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination).

The laboratory utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

14.1.3 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by

different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

14.1.4 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QA Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses. Procedures for establishing control limits are found in the SOP for Control Limits (ADM-CTRL_LIM).

14.1.5 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. the laboratory has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Subsampling and Compositing Aqueous and Soil Samples* (WET-SSMP) and the *SOP for Tissue Preparation* (WET-TISP). Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

14.1.6 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using the laboratory or project-specified data qualifiers.

14.2 Method Detection Limits, Method Reporting Limits, and Limits of Detection/Quantitation

Method Detection Limits (MDL) for methods performed at the laboratory is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

14.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

14.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

14.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

14.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

14.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

14.3.5 Initial Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in for calibration. Once it is determined that there is no defect or error in the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

14.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCALs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCALs analysis is as indicated in the method.

14.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

14.3.8 Labeled Standards

Labeled standards are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each labeled standard, and the recovery is a measurement of the overall method performance.

$$\boldsymbol{Recovery}\ (\%)\ =\ (\frac{\boldsymbol{M}}{\boldsymbol{T}})\ \boldsymbol{x}\ \mathbf{100}$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

$$\boldsymbol{Recovery}\ (\%) = (\frac{\boldsymbol{M}}{\boldsymbol{T}})\ \boldsymbol{x}\ \mathbf{100}$$

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

14.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water sample as required by state regulation.

14.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$\mathbf{Recovery\ (\%) = (S - A)\ x\ 100 \div T}$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

14.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the

method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where

S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

14.3.13 Control Charting

The generation of control charts is routinely performed at the laboratory. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements.

14.3.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at the laboratory undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at the laboratory; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

14.3.15 Measurement Uncertainty

Measurement uncertainty is associated with most of the results obtained in laboratory testing. It may be meaningful to estimate the extent of the uncertainty associated with each result generated by the laboratory. It is also useful to recognize that this measurement of uncertainty is likely to be much less than

that associated with sample collection activities. The uncertainty associated with the analytical measurement process can be estimated from quality control data. When requested, the laboratory provides uncertainty information as described in the SOP CE-QA010, *Estimate of Uncertainty of Analytical Measurements*. The estimation of uncertainty relates only to measurements conducted in the laboratory.

14.4 When data quality objectives or quality control measures are not met, due to the sample matrix or anomalies, incompatibility of the methodology and sample type, statistical outliers, random error, or other factors, it may be necessary to apply data qualifiers to reported data. A list of standard data qualifiers is given in Appendix H.

15) Control of Non-Conforming Environmental Testing Work

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events, such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using the NCAR database. The procedure and responsibilities for addressing nonconforming work is defined in SOP CE-QA008, *Nonconformance and Corrective Action.* Nonconformances are reported to the client using various means (i.e., voice, email, narrative, etc.). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The QA Manager periodically reviews all NCARs looking for chronic, systematic problems that require more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Results from non-conforming environmental testing work generally require the need for qualified data on analytical reports. A list of standard data qualifiers is given in Appendix H. Additionally, the report narrative will provide an example of the nonconformance and the impact on results.

16) Corrective Action, Preventive Action, and Improvement

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of calculations, a check of instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the QA Manager may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to

corrective action taken to prevent recurrence. Various preventive action processes are used for eliminating a potential problem, or averting a problem before it occurs. This is explained in SOP CE-QA008, *Nonconformance and Corrective Action*.

Preventive action is focused on using existing information or experiences to anticipate potential problems and eliminating likely causes of them. Preventive action is a pro-active process and tied to results from corrective action as well as opportunities for improvement. The laboratory uses preventive action processes to avoid errors and implement improvements. The SOP CE-GEN004, *Preventive Action*, describes procedures used. Examples of preventive action are given in this SOP. The laboratory also uses ideas from staff, client feedback, and other input mechanisms to identify potential improvements. The weekly staff meeting regularly includes reports on improvements made or underway.

16.1 Preventive Maintenance

Preventive maintenance is a crucial element of the QA program. Equipment and instruments are regularly maintained by qualified laboratory staff or under commercial service contracts. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at the laboratory contain extensive information about the instruments used at the laboratory, including:

- The equipment serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of the equipment when it was received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification, or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used by the laboratory. They may be found in the various SOPs for routine methods performed on an instrument and are found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the department supervisor. In the case of non-routine repair of capital equipment, the department supervisor is responsible for providing the repair either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each department maintains a critical parts inventory. This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as GC/MS jet separators and electron multipliers. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See Appendix E for a list of equipment and whether primarily maintained by laboratory or service providers.

17) Control of Records

The laboratory maintains a record system, which ensures that all laboratory records of analysis data are retained and available. Records may be in hard copy form or electronic, in the form of .pdf rendering of hard copies, or in databases. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the SOP HE-QA002, *Report, Data, and Record Archival.*

17.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analysis performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Standard ID number for standards used;
- Copies of report sheets submitted to the service request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary

Individual sets of analyses are identified by analysis data and service request number. Since many analyses are performed using computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the original record, if performed on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences, are kept using a separate documentation system. This system is used to archive data on a batch specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

18) Audits

Quality audits are an essential part of the Quality Assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitative evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

18.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of the laboratory are conducted regularly by various regulatory agencies and clients. Appendix J lists the certification and accreditation programs in which the laboratory participates. Programs and certifications can be added as necessary.

Internal system audits of the laboratory are conducted regularly under the direction of the QA Manager. The internal audit procedures are described in SOP CE-QA001, *Internal Audits*. The internal audits are performed as follows:

- System Audit – this is an annual audit of all operational areas in the laboratory to evaluate compliance with operational and technical procedures. Focus is on sample handling, preparation, analysis, and technically sounds practices. Three

primary concepts are 1) is the procedure in use the same as that described in the SOP, 2) the use of sound analytical techniques and practices, and 3) sample handling/preparation. Topics as calibration, instrument operation/maintenance, data interpretation, and reporting results are included. Hardcopy data and/or report audits may be included.

Process audits may be on larger audit event or a series of audits such that all areas of the laboratory are audited over a year. Process audits conducted over the four calendar quarters will follow the schedules listed in the audit plan.

- Electronic data audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, use of appropriate files, and other components of the analysis. Each applicable instrument is periodically audited by reviewing randomly selected data files.

All audit findings and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

18.2 Performance Audits

The laboratory participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in SOP CE-QA006, *Proficiency Sample Testing Analysis.* The laboratory routinely participates in the following studies:

- Water Pollution (WP), 2 per year.
- Water Supply (WS), 2 per year.
- Hazardous Waste/Soil (LPTP), 2 per year.
- Other studies as required for certifications, accreditations, or validations

PT Samples are processed by entering them into the LIMS system as samples and are processed the same as field samples (following the PT provider instructions). The laboratory departments handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory departments submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the PT samples and audits are reviews by the QAM, Technical Director, the laboratory staff, and the Manager of Quality Assurance, USA. For any results outside the acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

19) Management Review

An annual review of the laboratory's quality system and testing activities is conducted by the laboratory's management team to ensure the continuing suitability and effectiveness of the quality system and testing activities and to introduce any necessary changes or improvements. The review ensures that the quality system of the laboratory continues to conform to the requirements of the ISO 17025:2005 and various accrediting authorities, including NELAP/TNI.

General procedures for the review are described in SOP CE-QA005, *Laboratory Management Review*. When conducting the review, a standard list of items and categories is evaluated. The quality policies and their relation to testing activities are reviewed and any necessary changes are identified. The review also notes significant changes that have taken place or need to take place in the quality system and the organization, facilities, equipment, procedures and activities of the laboratory.

The review is documented by the laboratory QA Manager. Action items, including preventive actions and improvements, should be identified. Results should feed into the laboratory's planning process.

20) Personnel

20.1 Personnel Training

Job descriptions, including technical position descriptions are used for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources Department, and are available for review. In order to assess the technical capabilities and qualification of a potential employee, all candidates for employment are evaluated, in part, against the appropriate technical description.

Training begins on the first day of employment at the laboratory when the company policies are presented and discussed. Safety and Quality System requirements are integral parts of initial and ongoing training processes at the laboratory. Safety training begins with the reading of the ALS Environmental Health and Safety Manual. Employees are also required to attend periodic safety meeting where additional safety training may be performed by the Environmental, Health, and Safety Officer.

Quality Systems training begins with QA orientation for new employees, which includes ethics/data integrity introductory training, and reading the QA Manual. During the employee's first year, the employee attends additional core ethics training and further learns about the laboratory quality systems as they relate to specific job functions. Each employee participates in annual ethics refresher training.

Employees are responsible for complying the requirements of the QA Manual and QA/QC requirements associated with their function(s). The laboratory also encourages personnel to continue to learn and develop new skills that will enhance their performance and value to the company. Ongoing training occurs for all employees through a variety of mechanisms. The corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in SOP CE-QA003, *Training Policy*. A training plan includes a description of the step-by-step process for

training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

20.2 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the SOP for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" LCS), analysis of 4 consecutive LCS analyses with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
  - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
  - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
  - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 20-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

20.3 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

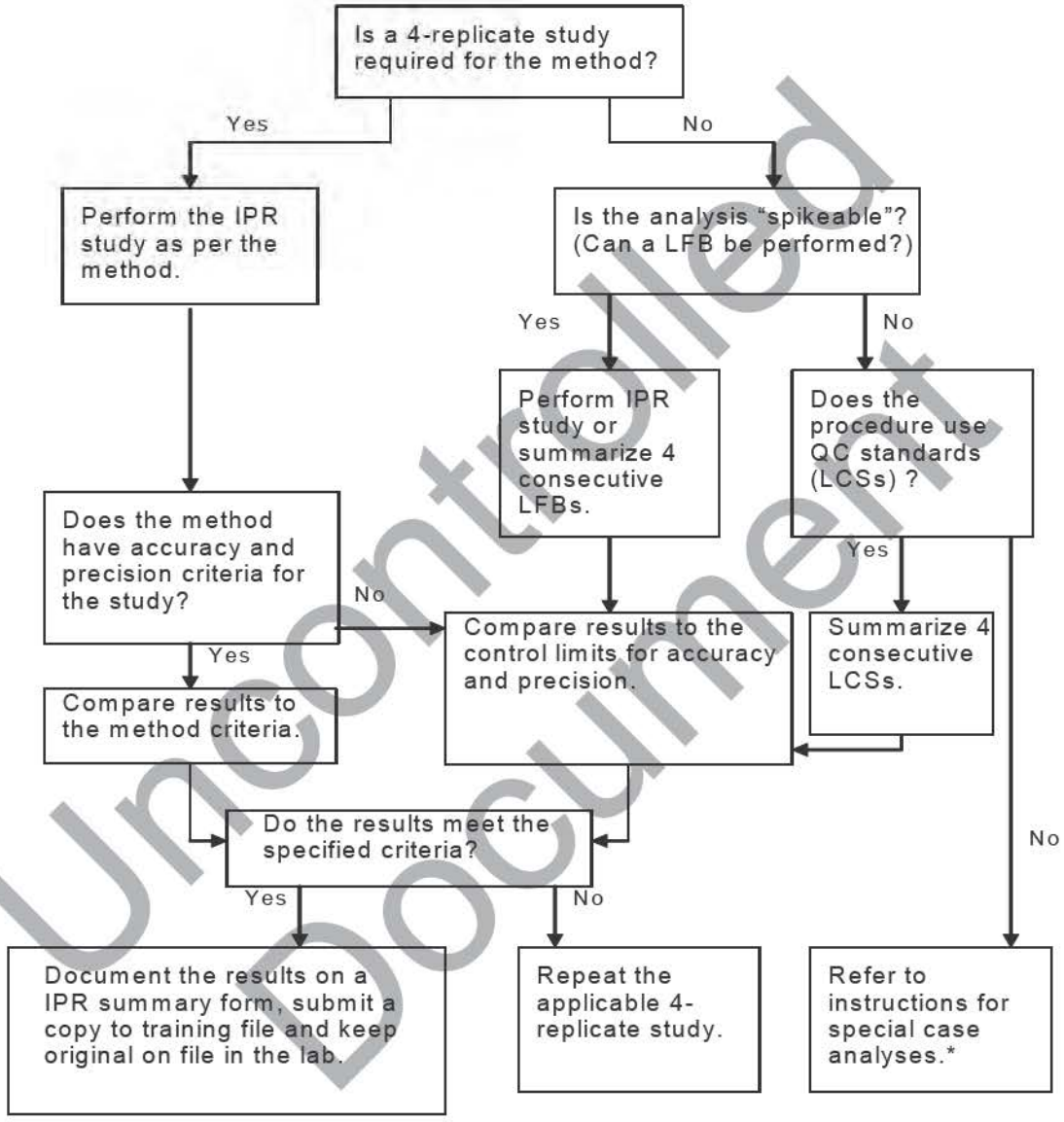
- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

20.4 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and internal resumes. The QA department maintains a record of the various technical skills and training acquired while employed by ALS. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in SOP CE-QA003, *Training Policy*.

Figure 20-1

Demonstration of Proficiency Flowchart



21) Reporting of Results

The laboratory reports the analytical data produced in its laboratories to the client via the Analytical Report. This report includes a transmittal letter, a case narrative, client project information, sample receipt and chain of custody information, specific test results, quality control data (as requested), and any other project-specific support documentation. The following procedures describe the procedures used for data reduction, validation and reporting.

21.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the raw data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the SOP CE-QA007, *Making Entries onto Analytical Records*.

The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report. Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the data and report hardcopy is forwarded to the supervisor or second qualified analyst who reviews the data. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Nonconformance and Corrective Action Report (NCAR) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed. Data review procedures are described in the SOP for *Laboratory Data Review Process* (ADM-DREV).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in SOP CE-QA002, *Manual Integration Policy* and SOP ADM-MI, *Manual Integration of Chromatographic Peaks*.

21.1.1 Validation of Results

The validity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures can include verifications of accuracy, QC samples, and system sensitivitycheck of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used

in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Initial Calibration – Following the analysis of calibration standards according to the applicable SOP the data is fit to an applicable and allowed calibration model (correlation coefficient, linear, average response factor, quadratic, etc.) and the resulting calibration is compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.
- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention

times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP for Confirmation Procedure for GC and HPLC Analysis (SOC-CONF). If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.

- Surrogate Results (Organic) – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – The LCS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.
- Matrix Spike Results – The MS percent recovery is calculated and compared to specified control limits. If the recovery is within control

limits the results are reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

21.1.2 Qualitative Data Evaluation

All sample results and QC results are reviewed to ensure correct identification of target analytes, when not inherent to the test method. Details particular to each analysis are given in the analytical SOP.

Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
  - The analyte must fall within the retention time window specified in the applicable SOP. The retention time window is established prior to analysis and documented.
  - For analyses all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis. Details for confirmation analysis are described in the SOP SOC-CONF, *Confirmation Procedures for GC and HPLC Analyses*. Confirmation Data Confirmation data will be provided as specified in the method.
  - When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS and LC/MS Methods - Two criteria are used to verify identification:
  - Elution of the analyte is at the same relative retention time (as defined by the method) as demonstrated in the standard.
  - The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.
  - When Tentatively Identified Compounds are to be reported for GC/MS, the spectrum for non-target peaks is compared to the current GC/MS reference library.

21.2 Data Reporting

It is the responsibility of each laboratory unit to provide the Project Manager with a final report of the data for each analysis, accompanied by signature approval. When the entire data set has been found to be acceptable, a final copy of the report is generated and approved by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package for the analysis is then placed into the service request file,

and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager.

When all analyses and departmental reports are completed the Project Manager reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A report narrative is written by the Project Manager to explain any unusual problems with a specific analysis or sample, etc. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is scanned and archived by service request number.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The SOP for *Data Reporting and Report Generation* (ADM-RG) addresses the flagging and qualification of data. The ALS-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the Project Manager to explain problems with a specific analysis or sample, etc.

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in SOP CE-QA010, *Estimation of Uncertainty of Analytical Measurements*.

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the client.

21.3 Deliverables

In order to meet individual project needs, the laboratory provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 21-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) deliverables for DoD QSM projects and state-specific drinking water formats.

When requested, the laboratory provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. The laboratory is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 21-1
Descriptions of ALS Environmental – Houston HRMS Standard Data Deliverables*
Tier I. Routine Analytical Report includes the following:
• Transmittal letter
• Chain of custody documents and sample/cooler receipt documentation
• Sample analytical results
• Method blank results
• Surrogate recovery results and acceptance criteria for applicable organic methods
• Dates of sample preparation and analysis for all tests
• Case narrative - optional
Tier II. In addition to the Tier I Deliverables, this Analytical Report includes the following:
• Laboratory Control Sample results with calculated recovery and associated acceptance criteria
• Matrix spike results with calculated recovery and associated acceptance criteria
• Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
• Case narrative - optional
Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:
• Case narrative - required
• Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits
• Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS are included.
Tier IV. Full Data Validation Package.
• All raw data associated with the sample analysis, including but not limited to: • Preparation and analysis bench sheets and instrument printouts, • For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive results, and the results and spectra of TIC compounds when requested.
• QC data
• Calibration data (initial, verification, continuing, etc.),
• Calibration blanks or instrument blanks (as appropriate to method).

* If a project QAPP or program reporting protocol applies the report will be presented as required for the project.

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
14	5/1/2014	R. Pierrot	Reverted back to the previous QA Manual format (see revision 11) Reformatted to current ALS style. Updated key personnel, Organization charts, and equipment. Updated and reorganized appendices. Removed remaining CAS references and updated Houston HRMS and corporate SOP references. Minor changes to text in several areas to improve readability and presentation, without changing concept.

23) References for Quality System Standards, External Documents, Manuals, and Test Procedures

The analytical methods used at ALS Environmental, Houston HRMS generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at ALS Environmental, Houston are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- TNI Standard – Environmental Laboratory Sector, Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis*, EL-V1-2009.
- Quality Standards. American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *DoD Quality Systems Manual for Environmental Laboratories,* Versions 4.2 and 5.0
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 5th Edition, EPA 815-B-97-001 (January 2005).
- *Procedure Manual for the Environmental Laboratory Accreditation Program,* Washington Department of Ecology, 10-03-048, September 2010.
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.

- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998) and SM On-Line. See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and EPA Method Update Rule 2007 and 2012.
- 40 CFR Part 141, National Primary Drinking Water Regulations and EPA Method Update Rule 2007.
- Analytical Methods for Petroleum Hydrocarbons, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
    - U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA-540/R-94/012 (February 1993).
- U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94/013 (February 1994).
- Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations (March 1982) and as Revised (July 1983 and April 1991).
- Identification and Listing of Hazardous Waste, California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater, EPA 821-R-93-017 (October 1993).
- Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

Internal program-level QA documents are listed in Appendix I.

24) Appendices

APPENDIX A – Glossary

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accreditation Body: The territorial, state or federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.

Accreditation Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

Analysis Date: The calendar date of analysis associated with the analytical result reported for an accreditation or experimental field of proficiency testing.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Bias: The systematic distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

Calibration Standard: A substance or reference material used for calibration.

Certified Reference Material (CRM): Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability to a national metrology institute.

Chain of Custody: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Field of Proficiency Testing (FoPT): Analytes for which a laboratory is required to successfully analyze a PT sample in order to obtain or maintain accreditation, collectively defined as: matrix, technology/method, analyte.

Finding: An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Holding Time: The specified maximum time that can elapse between two specified sampling and/or analytical activities.

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish evaluate accuracy and bias for associated sample analyses.

Legal Chain of Custody Protocols: Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect.

Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement System: A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States National Metrology Institute (NMI).

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Primary Accreditation Body (Primary AB): The TNI-NELAP accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.

Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Testing (PT): A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

Proficiency Testing Provider (PTP): A person or organization accredited by the TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.

Proficiency Testing Sample (PT Sample): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

Proficiency Testing Study (PT Study): A single complete sequence of circulation of proficiency testing samples to all participants in a proficiency test program.

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Control: The overall system of technical activities that continually measures the performance of a process, item, or service against defined standards to verify that they meet the stated requirements. Also, the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities.

Quality System Matrix: These matrix definitions be used for purposes of batch and quality control requirements:

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples are grouped according to type of tissue (i.e. marine vs. plant).

Chemical Waste: A product or by-product of an industrial process that results in a matrix not otherwise defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid, product, or solvent not miscible in water and with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis that records the original work steps, observations, and measurements, whether performed by an analyst or instrument. This documentation includes, but is not limited to field notes, electronic data, analysis bench sheets, run/injection logs, printouts, chromatograms, instrument outputs, and handwritten records for calibration, sample preparation, and sample analysis for field samples and QC samples.

Reference Material: Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Secondary Accreditation Body (Primary AB): A TNI-NELAP accreditation body responsible that accredits the laboratory based on the Primary AB accreditation and procedures.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): A written document that details the process for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the procedures for performing certain routine or repetitive tasks.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

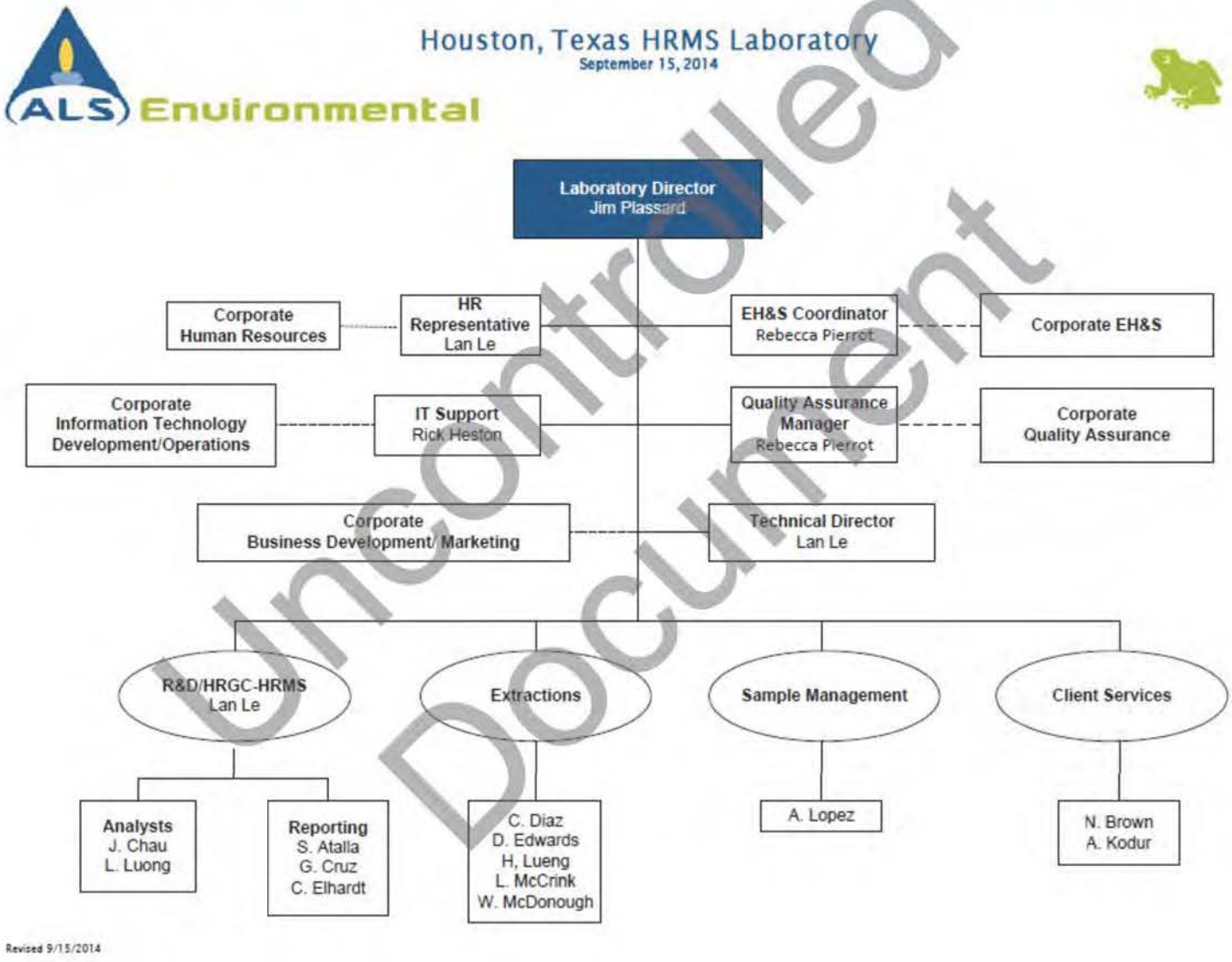
Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

Verification: Confirmation by examination and objective evidence that specified requirements have been met.

Acronyms

ASTM - American Society for Testing and Materials
A2LA - American Association for Laboratory Accreditation
CARB - California Air Resources Board
CAS - Number Chemical Abstract Service registry Number
CFC - Chlorofluorocarbon
CFU - Colony-Forming Unit
DEC - Department of Environmental Conservation
DEQ - Department of Environmental Quality
DHS - Department of Health Services
DOE - Department of Ecology
DOH - Department of Health
EPA - U. S. Environmental Protection Agency
ELAP - Environmental Laboratory Accreditation Program
GC - Gas Chromatography
GC/MS - Gas Chromatography/Mass Spectrometry
LOD - Limit of Detection
LOQ - Limit of Quantitation
LUFT - Leaking Underground Fuel Tank
M - Modified
MCL - Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL - Method Detection Limit
MPN - Most Probable Number
MRL - Method Reporting Limit
NA - Not Applicable
NC - Not Calculated
NCASI - National Council of the Paper Industry for Air and Stream Improvement
ND Not Detected
NIOSH - National Institute for Occupational Safety and Health
PQL - Practical Quantitation Limit
RCRA - Resource Conservation and Recovery Act
SIM - Selected Ion Monitoring
TNI – The NELAC Institute
TPH - Total Petroleum Hydrocarbons

APPENDIX B – Organization Charts, Key Personnel, and Report Signatories



Resumes of Key Personnel

Jim Plassard

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ALS Environmental

Education

Purdue University – West Lafayette, IN
BSc in Biology 1992

Affiliations

American Society of Mass Spectrometry

Laboratory Director

2012 - Present

Oversees all aspects of the HRMS operation. Ensures that quality and financial objectives are on target. Directs the laboratory in scheduling, prioritization, key improvements and optimizations, including staff development and training, as well as method development. Ensures continued growth of the laboratory through business development and client services.

PREVIOUS EXPERIENCE

SGS North America — HRGC/HRMS Director, '03 - 12
Wilmington, NC

Responsible for the coordination of analytical requirements for the HRMS laboratory including analysis of environmental samples by a variety of EPA methodologies, including 8290, 1613, 23, and 1668. Served as the primary point of contact for the HRMS group and as technical consultant to project managers and clients..

SGS North America — General Manager/Senior Analyst, '09 - 10
Wilmington, NC

In addition to above responsibilities, responsible for overall quality and financial performance of the laboratory. This included implementation of reorganization projects to improve efficiency with the primary objective of improving performance for clients. Areas of management included analytical chemistry, analysis production, project management, client services, data management, sample control, and safety,

AIT Laboratories — Interim Laboratory Director, '02 - 03
Indianapolis, IN

Responsible for quality control and quality assurance. Performed final review and approval of results and documentation prior to release to the client. Upgraded the QC program and initiated the Lab Safety program. Areas of management included QC, Chromatography, Screening and Method Development.

AIT Laboratories — Technical Director, '02 - 03
Indianapolis, IN

Directed the preparation and analysis of all chromatographic assays of forensic, clinical and drugs of abuse samples. Implemented improvements in quality control, assay validation, and procedure documentation. Guided the laboratory through ISO 9000-2000 accreditation. Implemented and managed a Method Development Team resulting in multiple new assays.

AIT Laboratories — Senior Analytical Chemist, '99 – '00
Indianapolis, IN

Performed various extractions and analyses of toxicological samples. Responsible for routine and non-routine maintenance of instrumentation including GCMS, LCMS, GC-FID, GC-NPD, GC-ECD, HPLC-UV, and HPLC-Fluorescence.

Pace Analytical — Low-Res Dioxin Analyst, '93 – '99
Indianapolis, IN, 1993

Responsibilities: Write job duties and responsibilities here. Can include number of people managed, projects completed, etc. Always leave an extra space after this paragraph to separate from the next job.

Lan Le, Ph.D

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ALS Environmental

Education

Kansas State University
Manhattan, KS
BS, Chemical Engineering, 1981

University of Houston
Houston, TX
Ph.D., Chemistry, 1987

Publications

Expert Systems for the Analytical Laboratory, Demonchy, A.R.; Aretteig, J.R.; Le, L.; and Deming, S.N., Analytical Chemistry, 60, 1355A, 1988.

Technical Director

2011 - Present

Performs competently with complex instruments and methods and is responsible for date interpretation, quality control and reporting. Plans, conducts, and supervises (as a lead) complex analyses using advanced instrumentation. Prepares standard operating procedures and specifications for processes and tests. Works with complex analytical systems, technical report writing, and client interface. Assists in training staff, analysts and technical assistance. Senior technical advisor for teams and projects.

Documentation of Demonstration of Capabilities is available for review.

PREVIOUS EXPERIENCE

Columbia Analytical Services, Inc. **Interim Laboratory Director**, '11-'12
Houston, TX
Oversees all aspects of the HRMS operation. Ensures that quality and financial objectives are on target.

Columbia Analytical Services, Inc. **Technical Manager II**, '08-'11
Houston, TX
Supervise staff of 13 scientist and technicians, oversee data collection & validation, manage calibration & maintenance of analytical instrument systems, method development, perform training. interface with project management in support of technical solutions for clients.

Southern Petroleum Laboratories **GC/MS Supervisor**, '93-'08
Houston, TX
Supervise staff of 13 scientist and technicians, oversee data collection and validation, manage calibration and maintenance of analytical instrument systems, method development, perform training, and interface with project management personnel in support of technical solutions for clients.

Core Laboratories **Organics Supervisor,** '91-'93
Houston, TX
Supervise Organics Laboratory including both GC/MS and GC analyses. Method interpretation and implementation. Supervised and performed both volatile and semivolatile organics determinations. Used HP-RTE data systems for data interpretation, quantitation, and reporting. Performed instrument maintenance and troubleshooting on HP 5970 GC/MS systems, autosamplers, integrators, and related analytical equipment.

MBA Laboratories **GC/MS Analyst**, '88-'90
Houston, TX
Performed environmental GC/MS analyses samples in accordance with EPA protocols using an HP 5970 GC/MS with an HP-1000 RTE data system.

Shell Development Company **Contract Chemist,** '87-'88
Houston, TX
Performed research in a group that developed an expert system for analyzing spectra obtained from a variety of samples, including polymers and heavy oils, which had undergone neutron irradiation.

Exxon Research and Engineering Company **Contract Chemist**, '86-'87
Baytown, TX
Worked in a research group that developed and expert system for data interpretation for GC/MS hydrocarbon analyses, including naphthas and kerosene.

University of Houston, Chemistry Dept. **Chemistry Researcher**, '83-'87
Houston, TX
Responsibilities: Developed and authored a Reversed Phase-HPLC method on neutral surfactants. Studied varying HPLC gradients and performance factors.

Rebecca Pierrot

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ALS Environmental

Education

University of Texas
Austin, TX
BS, Chemistry, 2003

TNI Mentoring Session
Determination of Detection and Quantitation Limits

Rockhurst University
Continuing Education Center
Advanced Microsoft Excel, January 2007

TNI Seminar:
Standard Methods: Theory and Application, August 2008

Rockhurst University
Continuing Education Center
Management Skills for First Time Supervisors, September 2008

TNI Seminar:
How to Use Qualified Data, August 2009

TNI Seminar:
The New TNI Laboratory Accreditation Standards, January 2010

Affiliations

The NELAC Institute
2008 – Present

TNI Accreditation Body
Committee Member,
2009 – Present

Texas Association of
Environmental Professionals
2010 – Present

Laboratory Accreditation
Bureau Technical Advisory
Group
2010 - Present

Quality Assurance Program/EH&S Manager

2011 - Present

As Quality Assurance Program Manager, lead Houston's Quality Assurance/Quality Control Program. Responsible for reviewing, approving and controlling the quality systems of ALS' HRMS laboratory. Facilitate the review and changes to laboratory SOPs and the QA Manual. Document training through DoCs and attestations. Manage PE samples and document adherence to standard operating procedures. Review analytical data, perform internal audits and assure compliance with external audit findings. Maintain state and federal certifications. Facilitate Quality Assurance and Ethics training. Prepare quarterly and annual quality reports to senior management. Facilitate managerial review of the Houston laboratory's Quality Assurance Program.

AS EH&S manager, responsible for the implementation of the Environmental Health and Safety program of ALS North America to this facility. Duties include accident investigation and incident review, maintenance of all safety-related equipment and documents, and performing safety audits and reporting results to management.

PREVIOUS EXPERIENCE

ALS Laboratory Group. **Quality Assurance Manager**, '09-'11
Houston, TX

Responsibilities: Responsible for the management and implementation of a NELAC-Accredited quality system for an international environmental laboratory. This encompasses the areas of document control, employee training and certification, review of routine QC studies, and internal auditing.

ALS Laboratory Group **Quality Assurance Assistant**, '05-'09
(Formerly e-Lab Analytical)
Houston, TX

Responsibilities: Assisted in the development and implementation of a NELAC-Accredited quality system for an international environmental laboratory. Additional duties included level IV data package preparation and review, root cause analysis, employee training and method development.

Southern Petroleum Laboratories **GC Analyst, '04-'05**
Houston, TX

Responsibilities: Responsible for the preparation, analysis, and reporting, of purgeable aromatics and gas range organics using GC-PID, -FID (dual column/detector). Maintained and troubleshot HP GC Instruments, including calibration, column and trap replacement, and auto-sampler calibration/maintenance. Relevant software includes TurboChrom for data acquisition and processing and a MS Access-based LIMS for reporting results.

Austin Energy **Power Plant Chemist - Intern**, '01-'03
Austin, TX

Responsibilities: Responsible for sample collection and analysis (pH, conductivity, and spectrophotometry), water treatment and purification (reverse osmosis and ion-exchange), environmental sample collection (routine and outfall), and various laboratory management duties including calibrations, monthly environmental reporting, chemical inventory and purchasing. Organized various training sessions focused on safety practices in the plant.

Nicole Brown

10450 Stancliff Rd., Suite 210 • Houston, TX 77099 • +1 713-266-1599

ALS Environmental

Education

University of Houston – Houston, TX
B.S. - Biology, 2007

Houston Community College, Houston, TX
A.S. – Biology/ Chemistry, 2004

Project Manager

2012 - Present

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements. Provide technical support to clients regarding laboratory application to HRGC/HRMS projects requiring knowledge of a wide-range of requirements including US EPA CLP, AFCEE, ACOE, NFESC, RCRA, CWA, SDWA, EU and CAA. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. Responsible for direct technical project management providing technical and regulatory interpretation assistance, as well as project organization of work received and reported by the laboratory.

PREVIOUS EXPERIENCE

ALS Laboratory Group. **Project Manager '11 – '12**
Houston, TX

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. Responsible for direct technical project management providing technical and regulatory interpretation assistance, as well as project organization of work received and reported by the laboratory.

Columbia Analytical Services, Inc. **Project Chemist, '10 – '11**
Houston, TX

Assure project details are understood by technical and administrative staff and that analytical reports and EDDs comply with established project requirements. Manage GC/HRMS projects requiring a wide-range of requirements including US EPA CLP, AFCEE, ACOE, NFESC, RCRA, CWA, SDWA, EU and CAA.

Columbia Analytical Services, Inc. **Analyst, '08– '10**
Houston, TX

Oversee operations in Extraction Laboratory and SMO. Primary responsibilities include sample login, extraction, cleanup, and final concentration. Schedule daily tasks for extraction analysts. Run Methods 1668A, 1613B, 8280, 8290, TO-9A and 23. Aid in continuous improvement of existing methods and development of new methods. Perform non-routine and complex technical assignments.

Columbia Analytical Services, Inc. **Analyst, '07 – '08**
Houston, TX

Run Methods 1668A, 1613B, 8280, 8290, TO-9A and 23. Perform extractions, sulfuric acid clean up, silica gel column clean up, and blow downs/transfers. Receive and log arriving samples into CAS LIMS. Prepare and ship client bottle kit orders

Baylor College of Medicine Human Genome Sequencing Center **Research Technician, '07 – '07**
Houston, TX

Assist in development of DNA sequencing technology using PCR and micro-titer plate technology.

Arthi Kodur

10450 Stancliff Rd., Suite 210 • Houston, TX 77099 • +1 713-266-1599

ALS Environmental

Education

St. Thomas University, Houston, TX
MBA Courswork

National University, San Diego, CA
MFS in Forensic Science 2007

Texas A & M, College Station, TX
BS in Genetics 2005

Project Manager

2011 - Present

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements. Provide technical support to clients regarding laboratory application to HRGC/HRMS projects requiring knowledge of a wide-range of requirements including US EPA CLP, AFCEE, ACOE, NFESC, RCRA, CWA, SDWA, EU and CAA. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. Responsible for direct technical project management providing technical and regulatory interpretation assistance, as well as project organization of work received and reported by the laboratory.

PREVIOUS EXPERIENCE

ALS Laboratory Group. **Group Leader, Extractions, '08-'11**
Houston, TX

Oversee operations in Extraction Laboratory and SMO. Primary responsibilities include sample login, extraction, cleanup and final concentration. Schedule daily tasks for extraction analyst. Run Methods 1668A, 1613B, 8280, 8290, TO-9A and 23. Aid in continuous improvement of existing methods and development of new methods. Perform non-routine and complex technical assignments. Also responsible for environmental health and safety issues, such as overseeing the solid, solvent and acid disposal.

Columbia Analytical Services, Inc. **Analyst III, '07– '08**
Houston, TX

Ran Methods 1668A, 1613B, 8280, 8290, TO-9A and 23. Performed extractions, sulfuric acid cleanup, silica gel column cleanup and blow-downs/transfers. Received and logged in arriving samples into LIMS. Prepared and shipped client bottle kit orders.

Yancy Life **Science Instructor, '06 – '08**
Stafford, TA

Educated children from pre K through 5th on grade basic science concepts.

Genetic Profiles Corporation **Lab Technician, '05 – '06**
San Diego, CA

Acce specimens, extraction PCR, gel electrophoresis and gel analysis.

APPROVED SIGNATORIES FOR FINAL ANALYTICAL REPORTS

ALS Environmental, Houston - HRMS

Brown, Nicole
Kodur, Arthi
Le, Lan
Pierrot, Rebecca
Plassard, Jim

Update: October 2014 Approved by: Jim Plassard, Laboratory Director

APPENDIX C
ALS Environmental Confidentiality Agreement

Confidentiality Agreement

The Confidentiality Agreement (the "Agreement") is entered into by and between ALS Laboratory Group (hereinafter referred to as the "Company") and _____ (hereinafter referred to as "Employee").

WHEREAS, employee is presently employed by the Company in a position in which Employee will receive and have access to confidential business information and other secrets of the Company, and shall, to the best of Employee's ability, assist the Company in improving and developing the products and services of the Company; and

WHEREAS, employee is desirous of continuing such employment and receiving such disclosures of confidential business information, and assisting the Company in improving and developing its products and services.

NOW, THEREFORE, in consideration of One Hundred and xx/100 Dollars ($100.00) paid to the Employee by the Company and in further consideration of Employee's continued employment by the Company, this Agreement being a condition therefore and ancillary thereto, and in further consideration of the benefits to Employee pursuant to the employment by the Company, the receipt and sufficiency of all such consideration being hereby acknowledged by Employee, it is agreed between the Company and Employee as follows:

1. Confidential Business Information. Employee recognizes and agrees that the Company has certain confidential business information, including, but not limited to, compilations of information, customer lists, customer data, records, specifications, and trade secrets, and related business methods and techniques, which confidential business information are used by the Company to obtain a competitive advantage over the Company's competitors who do not know or use this information. Employee further recognizes and agrees that the protection of such confidential business information against unauthorized disclosure and use is of critical importance to the company to maintain its competitive position and Employee therefore agrees that use of, or disclose to any other person or entity, except as authorized by the Company in writing, any of the confidential business information of the Company. Employee also agrees not to disclose to the Company or utilize on the Company's behalf, any of the trade secrets or other confidential information of any of the Employee's former employers.

2. **Return of Confidential Business Information.** Upon termination of his employment for any reason, employee shall promptly deliver to the Company all drawings, manuals, letters, photographs, tapes or video recordings, records of any kind, and all copies thereof, that may be in the possession of, or under the control of, Employee pertaining to the Company's employers.

3. **Assignment of Rights to Company.** Employee agrees to assist the Company in all possible ways in the discovery, perfection, and development of new ideas, inventions, discoveries, devices, and methods in processes, all for the benefit of the Company and as its exclusive property. Employee agrees to and does hereby assign, transfer, and convey to the Company, or at the written direction of the Company and which are made, developed or conceived by Employee, either solely or jointly with others, during Employee's employment with the Company, whether prior or subsequent to the signing of this Agreement, whether made, developed or conceived by Employee during or outside of regular working hours or on or away from the Company's premises or at Employee's expense, the expense of the Company or some other person or persons. At any time, the Employee shall execute such documents requested by the Company to confirm the rights of the Company in the ideas, inventions, discoveries, and devices, methods and processes referenced in this Section 3.

4. **Reasonableness of Covenants.** Employee specifically acknowledges and agrees as follow: (i) the covenants set forth in this Agreement are reasonable and necessary to protect the goodwill and the operations and business of the Company; (ii) the time duration of the covenants set forth in this Agreement and are reasonable and necessary to protect the goodwill and the operations and business of the Company; (iii) the geographical area limitations of the covenants set forth in this Agreement are reasonable and necessary to protect the goodwill and the operations and business of the Company; (iv) the covenants set forth in this Agreement are not oppressive to Employee and do not impose a greater restraint on Employee than is necessary to protect the goodwill and the operations and business of the Company.

5. **Remedies.** Employee recognizes that irreparable injury or damage will result to the business of the company in the event to the breach of any covenant contained in this Agreement and Employee therefore agrees that in the event of such breach on the part of the Employee, the Company shall be entitled, in addition to any legal or equitable remedies and damages available, to an injunction to restrain the violation

thereof by Employee and all other persons action for or on behalf of Employee. Any claim of Employee against the Company shall not prevent the Company from enforcing any provision of this agreement. Further, in the event legal action is necessary to enforce any of Employee's obligations hereunder and the Company prevails in such legal action, the Company shall be entitled to a recovery of its attorney's fees expended in such action.

6. **Reformation.** Whenever possible, each provision of this agreement shall be interpreted in such manner as to be effective and valid under applicable law; provided, however, incase any on or more of the provisions contained in this Agreement shall, for any reason, be held to be invalid, illegal, or unenforceable in any respect, such invalidity, illegality, or unenforceability shall no affect any other provision of this agreement, and this Agreement shall be construed as if such invalid, illegal, or unenforceable provision had never been contained herein. Should a court of competent jurisdiction declare any of the provisions of this Agreement unenforceable due to any restriction of duration, territorial coverage, scene of activity, or otherwise, in lieu of declaring such provisions unenforceable, the parties hereto expressly authorize the court, to the extent permissible by law, to revise or reconstruct such provisions in a manner sufficient to cause them to be enforceable.

7. **Affiliates.** This agreement, and Employee's obligations hereunder, shall apply to any confidential business information, formulas, recipes, patterns, devices, secret inventions, processes, compilations of information, materials, ingredients, customer lists, records, specifications and trade secrets of any affiliate of the Company. For the purpose of this Agreement, the "affiliate" means any person that, directly or indirectly, controls, or controlled by, or is under common control with, another person"; "person" means any individual, corporation, partnership, joint venture, limited liability company, association, joint stock company, trust, unincorporated organization or any other form of entity; and "control" means the power to direct or cause the direction of the management and policies of a person, directly or indirectly, whether through the ownership of voting securities by contract, or otherwise.

8. **Compelled Disclosure.** In the event that Employee is requested or required (by oral questions, interrogatories, requested for information or documents, subpoenas, civil investigative demand or similar process) to disclose any of the confidential business information of the Company, it is agreed that Employee will provide the Company with immediate notice of such request(s), so that the Company may seek an

appropriate protective order or, if appropriate, waive Employee's compliance with this agreement. Employee agreed that, if in the absence of a protective order or the receipt of a waive hereunder, Employee is nonetheless, in the reasonable opinion of Employee's counsel, legally compelled to disclose the confidential business information of the Company or else stand liable for contempt or suffer other censure or penalty, Employee may, after prior notice to the Company, disclose such the confidential business information of the Company to the extent legally required.

9. **Indemnity.** Employee agrees to indemnify and hold harmless the Company, and its directors, officers, employees, agents, and attorneys, from and after the date hereof, against any and all actions, causes of action, claims, suites, proceedings, demands, assessments, demands, settlement, judgment, damages, loses, costs, and legal and other expenses arising out of or resulting from the breach or failure of Employee to Company with any covenant or agreement made herein.

10. **Choice of Law: Waiver of Trial by Jury.** This Agreement shall be construed in accordance with, and governed for all purposes by the laws of the State of Texas and obligations and undertakings of each of the parties to this contract shall be performable at Houston, Harris County. TO THE EXTENT NOT PROHIBITED BY APPLICABLE LAW, THE PARTIES HEREBY KNOWLINGLY, VOLUNTARILY, AND INTENTIONALLY WAIVE ANY RIGHT TO TRIAL BY JURY THAT THE COMPANY OR EMPLOYEE MAY HAVE IN ACTION OR PROCEEDING, IN LAW OR IN EQUITY, IN CONNECTION WITH THIS AGREEMENT. EACH PARTY REPRESENTS AND WARRANTS THAT NEITHER PARTY HAS REPRESENTED, EXPRESSLY, OR OTHERWISE THAT IT WILL NOT, IN THE EVENT OF LITIGATION, SEEK TO ENFORCE THIS RIGHT TO JURY TRIAL WAIVER. EACH PARTY ACKNOWLEDGES THAT THE OTHER PARTY HAS BEEN INCLUDED TO ENTER INTO THIS AGREEMENT BY, AMONG OTHER THINGS, THE PROVISIONS OF THE WAIVER.

11. **Waiver.** No waiver of any provision of this Agreement shall constitute a waiver of any other provision of this agreement, nor such waiver constitute a waiver of any subsequent breach of such provision.

12. **Acknowledgement of Receipt.** Employee acknowledges a receipt of a copy of this Agreement, which has been executed in multiple copies, all executed copies of that shall be deemed originals.

13. **No Promise of Employment.** It is expressly agreed that this Agreement is not a promise of future employment.

14. **Assignment: Survival.** This agreement shall not be assignable by Employee. This agreement and the obligations of Employee hereunder, shall survive the termination of Employee's employment with the Company.

15. **Entire Agreement.** This Agreement entered into by the Company and Employee, embodies the entire agreement and understanding between the Company and the Employee relating to the subject matter hereof, and supersedes all prior agreements and understandings relating to the employment and compensation of the Employee and may only be amended by a written agreement signed by all parties hereto.

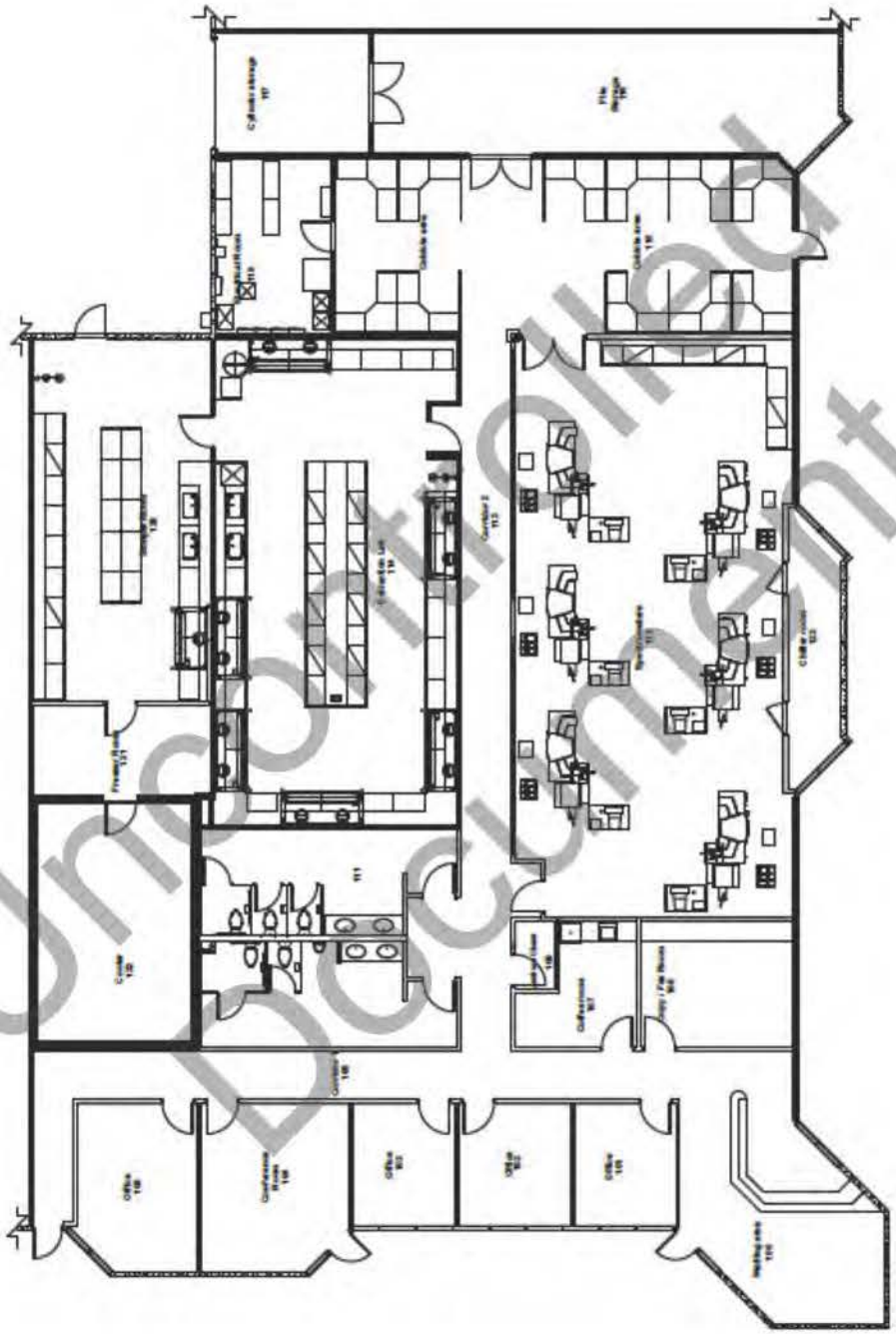
Employee Signature: _____ Date: _____

Employee Printed Name: _____

Witness: _____ Date: _____

Witness Printed Name: _____

APPENDIX D – Laboratory Floor Plan



APPENDIX E – Analytical Equipment

Table E.1 – Analytical Equipment

Department	Instrument Type	Instrument Make
Houston HRMS	HRGC/HRMS (E-HMS-01)	Agilent 6890N GC
		CTC A200S Autosampler
		Waters Autospect Ultima HRMS
	HRGC/HRMS (E-HMS-02)	Agilent 6890N GC
		CTC A200S Autosampler
		Waters Autospect Ultima HRMS
	HRGC/HRMS (E-HMS-03)	Agilent 7890A GC
		CTC A200S Autosampler
		Waters Autospect Premier HRMS
	HRGC/HRMS (E-HMS-04)	Agilent 7890A GC
		CTC A200S Autosampler
		Waters Autospect Premier HRMS
	Shaker	Eberbach Shaker Model 6000
	Rotary Evaporators	Buchi Rotovapor (R-200)/Waterbath (B-490)
		Buchi Rotovapor (R-201)/Waterbath (B-491)
		Buchi Rotovapor (R-201)/Waterbath (B-491)
	Nitrogen Evaporator	Zanntek Zip Vap #109A Turbovap LV
	Balance	Mettler Toledo PG603-S (Analytical)
		Ohaus Scout Pro
	Balance Calibration Weights	Ohaus 2000g
		Class S Weights
	Cold Storage	(3) refrigerators
		(5) Freezers
	Centrifuge	Clay Adams Dynac 101
	Drying Oven	VWR Model 1305U
	Muffle Furnace	LGO Element Furnace, BF51842-P-BC
	Homogenizer	Hobart HCM62
	Heating Mantles	Glascol 100D RJ30012 (4)
	DI Water System	Siemens ELGA Pure Lab Classic
	Sonicators	Branson Ultrasonic Cleaner (2510R-MT)
		Branson Ultrasonic Cleaner (5510R-MT)

Table E.2	Calibration And Maintenance Schedule – Houston Facility		
Instrument	**Activity**	**Frequency**	**Documentation**
Refrigerators, Freezers	Thermometers are immersed in liquid to the appropriate immersion line	Temperatures are recorded each day in use	Worksheet/logbook
GC/MS Systems Service Contract (Waters)	Ion gauge tube degassing Pump oil-level check Diffusion Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment – Tune MSD Auto sampler maintenance	As required Monthly Annually As required As required As required As required As required	Worksheet/logbook
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least annually	Worksheet/logbook
Deionized/ Water (Maintained by Siemens)	Check resistance Check deionizer light Replace cartridge & large mixed bed resins	Daily Daily As required	
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required	
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required	

Table E.3 Preventive Maintenance Procedures

Instrument	Activity	Laboratory Performed or Vendor Performed	Frequency
Refrigerators and coolers	Record temperatures Clean coils Check coolant	Laboratory Vendor Vendor	Daily Annually Annually
Fume Hoods	Face velocity measured Sash operation Change filters Inspect fan belts	Laboratory Laboratory Vendor Vendor	Quarterly As needed Annually Annually
Ovens	Clean Record Temperatures	Laboratory Laboratory	Annually Daily
Analytical Balances	Check alignment Check calibration Clean pans	Laboratory Laboratory Laboratory	Daily Daily After each use
High Resolution GC/MS	Check gas supplies Change septum Change injection port liner Clip first foot of capillary column Change guard column Replace analytical column Clean source Change pump oil	Laboratory Laboratory Laboratory Laboratory Laboratory Laboratory Laboratory Laboratory	Daily; replace when pressure reaches 50psi Daily As needed As needed As needed As needed As needed Annually

APPENDIX F – Containers, Preservation and Holding Times

Table F. 1 Containers Preservations and Holding Times

DETERMINATION	MATRIX[a, b]	CONTAINER[c]	PRESERVATION[d]	MAXIMUM HOLDING TIME[e]
Dioxins/Furans by EPA 8290	W	G	4°C	30 days
Dioxins/Furans by EPA 8280A	W	G	4°C	30 days
Dioxins/Furans by EPA 8290	S	G	4°C	30 days, 1 year if frozen
Dioxins/Furans by EPA 8280A	S	G	4°C	30 days, 1 year if frozen
Dioxins/Furans by EPA 1613B	W, DW	G	0-4°C	1 year
Dioxins/Furans by EPA 1613B	S	G	-20°C to -10°C	1 year
Dioxins/Furans by EPA 23	A	XAD	0-6°C	30 days
Dioxins/Furans by EPA 1613B	T	G	-20°C to -10°C	1 year
Dioxins/Furans by EPA 8290	T	G	-20°C to -10°C	30 days
Dioxins/Furans by EPA TO-9A	A	PUF	<4°C	7 days
Polychlorinated Biphenyls (PCBs) by EPA 1668	W	G	<6°C	1 year
Polychlorinated Biphenyls (PCBs) by EPA 1668	S	G	-20°C to -10°C	1 year
Polychlorinated Biphenyls (PCBs) by EPA 1668	T	G	-20°C to -10°C	1 year
Polychlorinated Biphenyls (PCBs) by CARB[f] 428	A	XAD	Ambient	45 days
Polycyclic Aromatic Hydrocarbons (PAHs) by CARB[f] 429	A	XAD	<4°C	21 days
Pesticides by EPA 1699	W	G	<6°C	7 days
Pesticides by EPA 1699	S	G	-20°C to -10°C	1 year

[a] A = Air, DW = Drinking Water, S = Soil or Sediment, T = Tissue, W = Water
[b] Amount of sample required: DW, W = 2x1L; S, T = 50g; A = contents of 1 trap
[c] G = Glass, PUF = Polyurethane foam plug, XAD = XAD filled glass trap
[d] NELAC §5.5.8.4(a)(1) – For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.
[e] Holding times listed for samples only. Extract holding times can be found in referenced technical methods.
[f] CARB = California Air Resource Board

APPENDIX G – Standard Operating Procedures

SOP Number	TITLE – Houston HRMS
CE-GEN001	Laboratory Ethics and Data Integrity
CE-GEN003	Records Management Policy
CE-GEN004	Preventive Action
CE-GEN005	Document Control
CE-GEN006	SOP for Data Recall
CE-GEN007	Procurement and Control of Laboratory Services and Supplies
CE-GEN008	Method Development
CE-GEN009	Establishing Standard Operating Procedures
CE-GEN010	Handling Customer Feedback
CE-GEN011	Assigning a TSR to a Project
CE-QA001	Internal Audits
CE-QA002	Manual Integration Policy
CE-QA003	Training Policy
CE-QA004	Qualification of Subcontract Laboratories
CE-QA005	Laboratory Management Review
CE-QA006	Proficiency Testing Samples
CE-QA007	Making Entries Onto Analytical Records
CE-QA008	Nonconformances and Corrective Actions
CE-QA009	Control Limits
CE-QA010	Estimation of Uncertainty of Analytical Measurements
CE-QA011	Performing Method Detection Limit Studies And Establishing Limits of Detection and Limits of Quantitation
CE-QA012	Quality of Standards and Reagents
HE-CHP	Chemical Hygiene and Safety Manual
HE-EXT001	Extraction of Liquid Samples for Analysis of PCB and PCDD/F Compounds by HRMS
HE-EXT002	Soxhlet Extraction of Solid/Tissue/Air Samples for Analysis of PCB and PCDD/F Compounds
HE-EXT003	Clean-up Processing of Sample Extracts Containing Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans, and Polychlorinated Biphenyls
HE-EXT004	Air Trap Preparation
HE-EXT005	SUBSAMPLING AND COMPOSITING OF AQUEOUS AND SOIL SAMPLES
HE-EXT006	Preparation of Standard Solution
HE-EXT007	Tissue Preparation
HE-EXT008	Total Solids
HE-GEN001	Washing Reusable Glassware and Equipment
HE-HMS001	Analysis of Polychorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry
HE-HMS003	HRMS DATA REVIEW AND REPORTING
HE-PM001	Project Management
HE-QA002	Report, Data, and Record Archival
HE-QA005	Calibration Verification of Support Equipment
HE-QA005	Local Addendum to the Corporate Procedure for LOD/LOQ

SOP Number	TITLE – Houston HRMS
HE-QAM	Houston HRMS Quality Assurance Manual
HE-SAF002	Exposure Control Plan - Bloodborn Pathogens
HE-SMO003	Waste Disposal
HMS-1668A	Method 1668A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment Biosolids and Tissue by HRGC/HRMS
HMS-1668B	Method 1668B: Chlorinated Biphenyl Congeners in Water, Soil, Sediment Biosolids and Tissue by HRGC/HRMS
HMS-1668C	Method 1668C: Chlorinated Biphenyl Congeners in Water, Soil, Sediment Biosolids and Tissue by HRGC/HRMS
HMS-8280A	Method 8280A: Analysis of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans by Gas Chromatography/High Resolution Mass Spectrometry (GC/HRMS)
SMO-BOT	Bottle Order Preparation and Shipping
SMO-VOLWARE	Checking Volumetric Labware
SMO-WET	Sample Receiving

APPENDIX H – Data Qualifiers

Data Qualifiers

HRMS Qualifier Set

- B Indicates the associated analyte was found in the method blank at >1/10th the reported value.
- E Estimated value. The reported concentration is above the calibration range of the instrument.
- H Sample extracted and/or analyzed out of suggested holding time.
- J Estimated value. The reported concentration is below the MRL.
- K The ion abundance ratio between the primary and secondary ions were outside of theoretical acceptance limits. Reported concentration is a conservative estimate, however EMPC correction was not applied.
- P Chlorodiphenyl ether interference was present at the retention time of the target analyte. Reported result should be considered an estimate.
- Q Monitored lock-mass indicates matrix-interference. Reported result is estimated.
- S Signal saturated detector. Result reported from dilution.
- U Compound was analyzed for, but was not detected (ND).
- X See Case Narrative.
- Y Isotopically Labeled Standard recovery outside of acceptance limits. In all cases, the signal-to-nois ratios are greater than 10:1, making the recoveries acceptable.
- i The MDL/MRL have been elevated due to a matrix interference.

APPENDIX I – Controlled and Normative Documents

Title	Revision Number or Date	Location*
EPA Method 1613	B - 10/94	H:\QAQC\Published Methods\
EPA SW-846 Method 8290	09/94,	H:\QAQC\Published Methods\
EPA SW-846 Method 8290A	A (2007)	H:\QAQC\Published Methods\
EPA SW-846 Method 8280	A - 12/96	H:\QAQC\Published Methods\
EPA Method 1668A	A - 08/03	H:\QAQC\Published Methods\
EPA Method 1668B	B - 11/08	H:\QAQC\Published Methods\
EPA Method 1668C	C - 04/10	H:\QAQC\Published Methods\
CARB Method 428	09/90	H:\QAQC\Published Methods\
CARB Method 429	07/97	H:\QAQC\Published Methods\
EPA Method TO-9	A - 01/99	H:\QAQC\Published Methods\
EPA Method 23	05/95	H:\QAQC\Published Methods\
EPA Method 1699	12/07	H:\QAQC\Published Methods\
EPA Method 1614	08/07	H:\QAQC\Published Methods\
EPA Method 1614A	A - 05/10	H:\QAQC\Published Methods\
NCASI Technical Bulletin 0551	05/89	H:\QAQC\Published Methods\
EPA SW-846 Method 8000	B - 12/96	H:\QAQC\Published Methods\
The NELAC Standard	06/03	H:\QAQC\Published Methods\
TNI Standard	09/09	H:\QAQC\Published Methods\
ISO/IEC 17025	2005	H:\QAQC\Published Methods\
EPA SOW DLM	2.2 - 12/09	H:\QAQC\Published Methods\
EPA SOW CBC	1.2 - 12/09	H:\QAQC\Published Methods\
EPA Manual for the Certification of Laboratories Analyzing Drinking Water	5th ed. - 01/05	H:\QAQC\Published Methods\
EPA NFG for Chlorinated Dibenzo-p-dioxins and Chlorinated Dibenzofurans Data Review	09/05	H:\QAQC\Published Methods\
Solutions to Analytical Chemistry Problems with Clean Water Act Methods	03/07	H:\QAQC\Published Methods\
UFP Workbook	1 - 03/05	H:\QAQC\Published Methods\
UFP for Implementing Environmental Quality Systems	2 - 03/05	H:\QAQC\Published Methods\

Title	Revision Number or Date	Location*
EPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1: Fish Sampling and Analysis	3rd ed. - 11/00	H:\QAQC\Published Methods\
Organization for Economic Co-operation and Development, Principles on Good Laboratory Practice	01/98	H:\QAQC\Published Methods\
DoD QSM	4.2 - 10/2010	H:\QAQC\Published Methods\
40 CFR 136 and 141	07/08	H:\QAQC\Published Methods\
A2LA P101 - Reference to A2LA Accredited Status - A2LA Advertising Policy	06/17/2011	H:\QAQC\Published Methods\
A2LA P102 - A2LA Policy on Measurement Traceability	05/05/2011	H:\QAQC\Published Methods\
A2LA P102a - A2LA Policy on Measurement Traceability for Life Sciences	10/18/2007	H:\QAQC\Published Methods\
A2LA P103 - Policy on Estimating Measurement Uncertainty for Testing Laboratories	11/06/2009	H:\QAQC\Published Methods\
A2LA R103 A2LA General Requirements for Proficiency Testing	11/06/2009	H:\QAQC\Published Methods\Regulations\A2LA R103.pdf

Page 6 - Accreditation Program Requirements

State/Agency	Location (website)
A2LA	http://a2la.org/
Arizona	http://www.azdhs.gov/lab/license/env.htm
Arkansas	http://www.adeq.state.ar.us/techsvs/labcert.htm
California	http://ww2.cdph.ca.gov/certlic/labs/Pages/ELAP.aspx
EPA ASB	http://www.epa.gov/superfund/programs/clp/index.htm
Florida	http://www.floridadep.org/labs/qa/dohforms.htm
Illinois	http://www.epa.state.il.us/labs/programinfo.html
Louisiana ELAP	http://www.deq.louisiana.gov/portaltrait
Louisiana DHH	http://www.dhh.louisiana.gov/offices/page.asp?id=250&detail=8332
Maine	http://www.maine.gov/dhhs/eng/water/Templates/LabCertification/LabCertification.htm
Michigan	http://www.michigan.gov/deq/0,1607,7-135-3307_4131_4156---,00.html
Minnesota	http://www.health.state.mn.us/divs/phl/cert/index.html
Nevada	http://ndep.nv.gov/bwqp/lab/labservice.htm
New Jersey	http://www.state.nj.us/dep/oqa/labcert.html
New Mexico	http://www.nmenv.state.nm.us/dwb/index.htm
New York	http://www.wadsworth.org/labcert/elap/elap.html
Oklahoma	http://www.deq.state.ok.us/csdnew/labcert.htm
Oregon	http://www.deq.state.or.us/lab/ORELAP/orelap.htm
Pennsylvania	http://www.depweb.state.pa.us/labs/cwp/view.asp?a=3&Q=515609&labsNav=|
Tennessee	http://www.state.tn.us/environment/dws/drinking_water_program.shtml
Texas	http://www.tceq.state.tx.us/compliance/compliance_support/qa/env_lab_accreditation.html
Utah	http://health.utah.gov/lab/labimp/
Washington	http://www.ecy.wa.gov/
West Virginia	http://www.wvdep.org/item.cfm?ssid=11&ss1id=166
Wisconsin	http://www.dnr.state.wi.us/org/es/science/lc/APPLICATION/Index.htm

APPENDIX J – Laboratory Accreditations

Accrediting Body	Certificate Number
DoD ELAP	2897.01
ISO 17025	2897.01
Arizona	AZ0725
Arkansas	12-035-0
California	2452
Florida/NELAP	E87611
Hawaii	N/A
Illinois/NELAP	003004
Kansas/NELAP	E-10406
Louisiana/NELAP	03048
Louisiana/NELAP	LA120014
Maine	2012017
Maryland	343
Michigan	9971
Minnesota	048-999-427
Nevada	TX014112013A
New Jersey	TX008
New Mexico	N/A
New York/NELAP	11707
Oklahoma	2012-133
Oregon/NELAP	TX200002-009
Pennsylvania/NELAP	004
Tennessee	TN04016
Texas/NELAP	T104704216-12-3
UTAH/NELAP	TX014112013-2
Soil Import Permit	P330-12-00002
Washington/NELAP	C819-12
West Virginia	347

SOP ADDENDUM FORM

DATE: 6/11/2015	SOP TITLE: PCCC/F by ~~HRSM~~ HRMS (QLC 6/12/2015)
SOP #: HE-~~HSM~~HMS-001 (QLC)	DEPARTMENT: Instrumentation
REV #: 0.0	SUPERVISOR:

		Date
Reviewed by Supervisor:	[signature]	7/6/15
Reviewed by QA Manager:	[signature]	6/12/2015
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Approved changes to the specified document

The following sections correct information contained in, and shall be used in lieu of the corresponding section of the current SOP revision. These changes will be incorporated into the next revision of the referenced SOP.

Section	Revision
13.3.2.2	0

SOP cites the recovery for the low-level LCS at 70 – 130%. the laboratory low-level standard recovery can be 50 – 150% for Arizona.

23	0

Include the reference to the Manual for the Certification of Laboratories Analyzing Drinking Water, fifth Ed.

This SOP was effective 02/01/2014 as indicated
on the signatures on page 2 of this document
RP 10/8/2014

ALS Standard Operating Procedure

DOCUMENT TITLE: *ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXINS AND POLYCHLORINATED DIBENZOFURANS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)*

REFERENCED METHOD:	*EPA 1613B / EPA 8290A / M23 / TO-9A*
SOP ID:	*HE-HMS001*
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ALS Environmental

ANALYSIS OF POLYCHLORINATED DIBENZO-P-DIOXINS AND POLYCHLORINATED DIBENZOFURANS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/HRMS)

EPA 1613B / EPA 8290A / M23 / TO-9A

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Approved By: _____ Date: 01-21-14

Technical Director – Lan Le

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Approved By: _____ Date: 01.21.14

Laboratory Director – Jim Plassard

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1) SCOPE AND APPLICATION

1.1 This procedure provides the analytical conditions and procedures for the detection and quantitative measurement of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in extracts by isotope dilution High Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS).

1.2 The procedure incorporates the analysis of PCDDs/PCDFs via the following referenced methods:

1.2.1 US EPA Office of Water, Method 1613B
1.2.2 US EPA Office of Solid Waste, SW846 Method 8290A
1.2.3 US EPA Office of Research and Development Method TO-9A
1.2.4 US EPA Office of Air Quality Planning & Standards Method 23

1.3 The following compounds can be determined by this method:

Analyte	CAS Registry No
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin (PeCDD)	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin (HxCDD)	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin (HxCDD)	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin (HpCDD)	35822-46-9
1,2,3,4,6,7,8,9-Octachlorodibenzo-*p*-dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)	39001-02-0
Total Tetrachlorodibenzo-*p*-dioxin (TCDD)	41902-57-5
Total Pentachlorodibenzo-*p*-dioxin (PeCDD)	36088-22-9
Total Hexachlorodibenzo-*p*-dioxin (HxCDD)	34465-46-8
Total Heptachlorodibenzo-*p*-dioxin (HpCDD)	37871-00-4
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3

1.4 The calibration range of this procedure is 0.25 to 200 ng/mL for TCDD/TCDF and

PeCDD/PeCDF. This equates to 5 to 4,000 ppq for a 1L water samples, 0.5 to 400 ppt for a 10g soil, sediment, ash, or tissue samples, and 5 to 4000 pg/sample for air samples (TABLE 1).

1.5 A calculation for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to obtain a total toxicity equivalence quotient (Total TEQ) is described. Refer to the current revision of HE-HMS003 for further discussion of Total TEQ.

1.6 This procedure is designated for use by, or under the supervision of analysts experienced with residue analysis and skilled in HRGC/HRMS.

1.7 This procedure details the operating conditions and requirements for PCDD/PCDF analysis by HRMS. For specific details regarding the instrument operations and troubleshooting, refer to the User Manual for each component of the system (Section 19)).

2) METHOD SUMMARY

2.1 Samples are prepared for analysis using matrix specific extraction techniques, analyte specific clean up, and HRGC/HRMS analysis techniques. The SOPs for these procedures are listed in TABLE 1.

2.2 The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20µL of a nonane solution, containing 100 pg/µL of the recovery standards ^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,9-HxCDD, to the extract. These compounds are used to determine the percent recoveries of the isotopically (^{13}C) labeled standards. TABLE 2 outlines the relationships between recovery standards and labeled internal standards

2.3 A 1-µL aliquot of the concentrated extract is injected into a HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.4 The congeners associated directly with ^{13}C labeled standards are identified based on the elution of the congeners at the same retention times as their respective labeled standards. The remaining congeners for which ^{13}C labeled standards are not present are identified when their retention times fall within the windows established during routine calibration. All congeners are also identified based on the simultaneous detection of the two most abundant ions as well as the comparison of the ratio of these two ions with theoretical ion abundance ratios.

2.5 Quantitation of the individual congeners, total PCDDs, and total PCDFs is achieved in conjunction with the establishment of a multipoint calibration curve for each homologue. For details on processing data obtained through this procedure, refer to the current revision of the SOP for HRMS Data Review and Reporting (***HE-HMS003***).

3) DEFINITIONS

3.1 Abbreviations

PCDD	=	Polychlorinated dibenzo-*p*-dioxin
PCDF	=	Polychlorinated dibenzofurans
TCDD	=	Tetrachlorodibenzo-*p*-dioxin
PeCDD	=	Pentachlorodibenzo-*p*-dioxin

HxCDD	=	Hexachlorodibenzo-*p*-dioxin
HpCDD	=	Heptachlorodibenzo-*p*-dioxin
OCDD	=	Octachlorodibenzo-*p*-dioxin
TCDF	=	Tetrachlorodibenzofuran
PeCDF	=	Pentachlorodibenzofuran
HxCDF	=	Hexachlorodibenzofuran
HpCDF	=	Heptachlorodibenzofuran
OCDF	=	Octachlorodibenzofuran
PFK	=	Perfluorokerosene
CS	=	Clean up standard
IS	=	Internal standard
RS	=	Recovery standard
HRGC	=	High-resolution gas chromatography
HRMS	=	High-resolution mass spectrometry
TEF	=	Toxicity equivalence factor
TEQ	=	Toxicity equivalent

3.2 **Polychlorinated dibenzo-p-dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs)** – compounds that contain from 1 to 8 chlorine atoms, resulting in a total of 75 PCDDs and 135 PCDFs. The structures are shown in Figure 1.

3.3 **Isomer** – compounds having the same number and type of chlorine atoms, but substituted in different positions.

3.4 **Internal Standard Solution**– a solution containing the isotopically labeled analogs that is added to all samples, including method blanks and quality control samples, before extraction. The labeled standards are used along with response factors to measure the concentrations of the analytes. (Table 2).

3.5 **Calibration Solutions** – solutions containing known amounts of unlabeled PCDDs/PCDFs and labeled standards (Table 4). These solutions are used to determine the instrument response of the unlabeled analytes relative to the C^{13} labeled standards.

3.6 **Recovery Standard Solution** – a solution containing the recovery standards that are added to the extract after final concentration for HRGC/HRMS analysis to determine the recovery efficiencies achieved for the C13 labeled standards (Table 3).

3.7 **Clean Up Standard Solution** – a solution containing one Cl37 labeled standard that is to be spiked to the sample extract prior to performing any clean up procedures (Table 3)

3.8 **Matrix Spike Solution** – a solution of native (unlabeled) PCDDs and PCDFs that are spiked into the laboratory control samples or matrix spike samples prior to extraction (Table 3).

3.9 **Method Blank (MB)** – represents the background contributions from glassware, extraction and clean up solvents. An MB is spiked with a solution of C13 labeled

internal standards, extracted, cleaned up, and analyzed by HRGC/HRMS in exactly the same manner as the test samples.

3.10 **Laboratory Control Sample** – A known matrix spiked with analytes representative of the target analytes. This sample is used to document laboratory performance.

3.11 **Allowable Marginal Exceedance** – random outliers to acceptance criteria, which are not occurring repeatedly, and a systematic way. The number of allowable marginal exceedances for any LCS/DLCS is 1, based on the 17 analytes reported for this procedure.

4) INTERFERENCES

4.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All materials used during the extraction procedure must be demonstrated to be free from interferences under the conditions of analysis by performing laboratory method blanks. Analysts should avoid using polyvinylchloride gloves.

4.2 The use of high purity reagents and solvents helps minimize interference problems. Solvent lots are tested for the absence of PCDDs and PCDFs prior to use by the laboratory.

4.3 Interferences co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated alkyldibenzofurans that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes are verified using reference standards. These values must correspond to the established retention time windows. Refer to HE-EXT003 for cleanup options available to reduce interferences, and to achieve lower detection limits.

4.4 Interference with 2,3,7,8-TCDF is well documented on the DB-5 chromatographic column. As such, all positive 2,3,7,8-TCDF detections are confirmed on a DB-225 (Section 12.6.3) chromatographic column which is capable of resolving 2,3,7,8-TCDF from other TCDF isomers.

5) SAFETY

5.1 The 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in positions 2,3,7,8 are known to have toxicities similar to that of 2,3,7,8-TCDD. As such, 2,3,7,8-chlorine substituted isomers can pose health hazards if handled improperly. Only highly trained individuals who are thoroughly versed in appropriate laboratory procedures and familiar with the hazards of dioxins should handle these substances.

5.2 The laboratory will ensure that all analysts receive adequate safety training prior to working with the chemicals and compounds associated with this method.

5.3 The solvents and acids in use in the laboratory may be hazardous and should be treated as such. Exposure to these chemicals must be kept to a minimum. See the

Environmental Health and Safety Manual, ***CHP*** (Chemical Hygiene Plan), Section 5, for more information. Additionally, material safety data sheets (MSDS) are available in the laboratory for review prior to handling solvents and reagents.

5.4 Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous due to the potential for inhalation and ingestion. Samples of this nature should be processed under a fume hood. Additionally, fitted masks, with charcoal filters should be worn to prevent the inhalation of dust. Any activity that may generate airborne contamination must be performed with good ventilation.

5.5 The toxicity or carcinogenicity of each reagent used in this method is not precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be kept to a minimum.

5.6 A useful method for determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper, extract the filter paper and analyze the extract.

5.7 Materials known, or suspected to contain 2,3,7,8-Substituted PCDD/PCDFs, including environmental samples submitted for analysis must be confined to designated work areas. This includes, but is not limited to samples, standards, reagents, and glassware.

5.8 Personal Protective Equipment (PPE) - Disposable gloves, lab coats, safety glasses, and fume hoods adequate for work with these materials must be used. Cut resistant liners may be worn to protect against injury caused by broken glass. At the option of the analyst, protective shoe covers and pants may be worn. See the Environmental Health and Safety Manual, ***CHP*** (Chemical Hygiene Plan), for more information.

5.9 Thorough washing of hands and forearms after each handling of samples or extracts and before breaks (coffee, lunch, and shift) is recommended. Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces. Gloves must be one time use only and must be changed frequently to minimize exposure.

5.10 Care must be taken when handling compressed gas tanks in the laboratory. All cylinders must be properly secured at all times. See Section 6 of the ***CHP*** for more information.

5.11 Low-level contamination is always a possibility for HRGC/HRMS analysis due to the chemical properties of dioxins/furans and the low detection limits of the instruments.

6) SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Refer to the appropriate preparatory SOP (TABLE 1) for sample collection, containers, preservation and storage requirements.

6.2 Sample extracts are stored in the dark at room temperature. Sample extracts must be analyzed within 45 days of extraction. Extract of tissue samples submitted for analysis by method 8290A must be analyzed within 45 days of *collection*. If analysis occurs after this holding time, the results are considered minimum concentrations, and must be noted as such in the case narrative.

6.3 Unused portions of sample extracts are stored for one year after analysis.

7) APPARATUS AND EQUIPMENT

7.1.1 **High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS)** – The laboratory utilizes HRGC/HRMS/Data Systems designed to meet the requirements for the analysis of PCDD/PCDFs.

7.1.2 Specifically, the laboratory employs the following systems:

7.1.2.1 An Agilent 6890N Gas Chromatograph with a CTC A200S Autosampler interfaced to a Micromass Autospec Ultima High Resolution Mass Spectrometer.

7.1.2.2 An Agilent 6890N GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Ultima HRMS.

7.1.2.3 An Agilent 7890A GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Premier HRMS.

7.1.2.4 An Agilent 7890A GC with a CTC A200S Autosampler interfaced to a Micromass Autospec Premier HRMS.

7.1.3 Micromass MassLynx (Version 4.1) is used to obtain all data from the HRMS system.

7.1.4 Chromatographic data is processed using OpusQuan version 3.6. Detailed procedures for data reduction are discussed in the current revision of ***HE-HMS003***.

7.2 GC Columns

7.2.1 The laboratory employs the following chromatographic columns capable of meeting method performance criteria outlined in Section 13.2.

7.2.1.1 60m DB-5 fused silica capillary column (J&W Scientific, Agilent 122-5062 or equivalent) – This column is used for the initial determination of PCDDs/PCDFs (TABLE 2).

7.2.1.2 30m DB-225 fused silica capillary column (J&W Scientific, Agilent 122-2232, or equivalent) – This column is used to confirm the presence of 2,3,7,8-TCDF in samples when the congener is detected on the DB-5 column. This column is also available for samples requiring 2,3,7,8-TCDD reporting only.

7.3 **Miscellaneous Equipment and Materials** – The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method (laboratory specific equipment in parentheses).

7.3.1 20mL scintillation vials (VWR, 66022-004)
7.3.2 Laboratory fume hoods
7.3.3 Pipet tips, 2-200uL and 10-1000uL (Eppendorf, 022492039 and 022492055, respectively)
7.3.4 GC Gooseneck Splitless Liners, 2mm inner diameter (Restek, 20797)
7.3.5 Thermogreen™ LB-2 11mm Septa (Supelco, 20654)
7.3.6 Autosampler Vial Caps, Seal with PTFE Liner, 11mm (Supelco, 27102-U)
7.3.7 Carrier Gas, Ultra High Purity Helium (Airgas)
7.3.8 Perfluorokerosene (PFK) (SynQuest, SpectraSynQ PKF-H, PN 1200-2-20)
7.3.9 Ultrasonicator (Bransonic Ultrasonic Cleaner, 5510R-MT)
7.3.10 Supeltex™ M-2A Ferrules 0.4mm ID (Supelco, 22474)

8) STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 All standards, reagents, and consumable materials must be logged in and labeled with a unique standard ID upon receipt. Refer to the current revision of the SOP for Standard Receipt and Preparation, ***HE-EXT006***, for details of standard handling and

traceability. Store standards in the dark at room temperature.

8.2 Solvents

8.2.1 Methanol - 99.9% minimum Assay (HPLC Grade, JT Baker, UN1230, or equivalent)

8.2.2 Nonane - 99.9% minimum Assay (HPLC Grade, Sigma, N29406-500mL, or equivalent)

8.2.3 Toluene - 99.9% minimum Assay (HPLC Grade, JT Baker, UN1294, or equivalent)

8.3 **High-Resolution Concentration Calibration Solutions** - A series of nonane solutions containing unlabeled and carbon-labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges of these series are homolog dependent. Refer to TABLE 4 for specific homolog concentrations at each calibration level. These standards are purchased prepared and are assigned the manufacturer's expiration dates. Store at room temperature in the dark.

8.4 **Window Defining/GC Column Performance Mix Solution (WDM)** - This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The labeled 2,3,7,8-TCDD and TCDF are also present (TABLE 5). This is purchased prepared at 200ng/mL and is assigned the manufacturer's expiration date. Store at room temperature in the dark.

8.5 **Recovery Standard Solution** - Individual labeled analytes are combined to make a recovery standard stock solution. Perform an additional dilution on the stock solution to make a recovery standard working solution. Store at room temperature in the dark.

8.6 **Initial Calibration Verification Standard (Reference Standard)** - This standard is a CS-3 mix purchased from a supplier other than the primary standard supplier, and serves as the second source verification of the initial calibration. This CS-3 mix is not used as the daily calibration verification standard. This standard is purchased pre-made and assigned the manufacturer's expiration date. Store at room temperature, in the dark.

8.7 **Research standards must be adequately labeled as such.** All labels for standards designated for research must also state specifically the intended research to be done, along with an appropriate qualifier, such as "For research purposes only."

9) PREVENTIVE MAINTENANCE

9.1 For details on performing preventive and routine maintenance, refer to the user manual for the specific component being maintained.

9.2 Record all maintenance activities in the designated instrument maintenance logbook.

9.3 Carrier gas - Inline purifiers or scrubbers must be in place for all sources of carrier gas. These remove water, oxygen, and hydrocarbons. Purifiers are changed as recommended by the supplier.

9.4 Gas Chromatograph

9.4.1 Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum,

column ferrule, and auto sampler syringe as needed. Liners are replaced, at a minimum, before every 12-hour shift. Seals changed when recent sample analyses predict a problem with chromatographic performance. Liners may be cleaned and re-used.

9.4.2 Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column-cutting tool.

9.4.3 The performance of the column will deteriorate over time. The rate of deterioration is also dependant on the nature of the samples analyzed. A new column is required when, despite preventive maintenance, acceptable routine and/or initial calibrations cannot be achieved.

9.5 Mass Spectrometer

9.5.1 Tune the MS as needed to result in consistent and acceptable performance.

9.5.2 The outer source should be cleaned, as needed, depending on the performance of the instrument.

9.6 Preventive maintenance is performed annually by the service engineer for instruments under a service contract.

10) RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to the instructions in this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are only to be performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. The department supervisor/manager or designee performs final review and sign-off of the data. Problems that require corrective action are to be documented by the analyst, and brought to the attention of the Operations Manager.

11) PROCEDURE FOR SAMPLE PREPARATION

11.1 Refer to the appropriate preparatory SOP as outlined in TABLE 1, for details regarding the preparation, extraction concentration and clean-up of samples.

12) PROCEDURE FOR HRGC/HRMS ANALYSIS AND CALIBRATION

12.1 Typical Chromatographic/Mass Spectrometric Conditions and Data Acquisition Parameters, actual parameters may vary based on instrument optimization.

Gas Chromatograph - Operating Conditions
Column coating: DB-5
Film thickness: 0.25µm
Column dimension: 60 m x 0.25mm
Injector temperature: 300°C
Splitless valve time: 1 min
Interface temperature: 300°C
Temperature program:

DB-5	INITIAL	INITIAL HOLD TIME,	TEMPERATURE RAMP,	FINAL TEMP	FINAL HOLD TIME,

STAGE	TEMP °C	MIN	°C/MIN	°C	MIN
1	150	5	35	215	5
2			1.5	230	6
3			7	315	5

Column coating: DB-225
Film thickness: 0.25µm
Column dimension: 30 m x 0.25mm
Injector temperature: 240°C
Splitless valve time: 1 min
Interface temperature: 320°C
Temperature program:

DB-225 STAGE	INITIAL TEMP °C	INITIAL HOLD TIME, MIN	TEMPERATURE RAMP, °C/MIN	FINAL TEMP °C	FINAL HOLD TIME, MIN
1	130	2.5	40	170	0
2			5	210	1
3			3	230	13

12.1.1 Mass Spectrometer (MS) Resolution

12.1.1.1 The mass spectrometer is operated in electron ionization mode, utilizing select ion monitoring (SIM) to monitor the ions listed in TABLE 10 for each of the five SIM descriptors. The total cycle time (including the voltage-reset time) is < 1 second. The set of ions monitored for the initial calibration is monitored during sample analysis. The lock-mass ion for each descriptor is defined in TABLE 10.

12.1.1.2 Details for tuning the instrument are outlined in the user manual for each instrument. The mass spectrometer tuning conditions are based on the groups of monitored ions shown in TABLE 10. By using a PFK molecular leak, tune the instrument to meet the minimum required static resolving power of 10,000 (10% valley) at m/z 304.9824 (PFK).

12.1.1.3 Using peak matching conditions and the aforementioned PFK reference peak, verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low mass and high mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors.

12.1.1.4 Additionally, for all methods except EPA 8290A, 3-5 masses from each descriptor must be monitored to verify 10,000 resolution is met. EPA 8290A requires that only m/z 304.9824 and 380.9760 meet the 10,000 resolution.

12.1.1.5 The chromatography time for PCDDs/PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, mass drift correction is required. This is why a lock-mass ion from the PFK reference compound is used to tune the instrument.

12.1.1.6 During tuning the level of PFK metered into the ion chamber during analysis, should be adjusted so that the amplitude of the most intense selected lock-mass ion signal does not exceed 10% of the full scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

Note: Excessive PFK may cause noise problems and contamination of the ion source resulting in an increase in downtime for source cleaning.

12.1.1.7 Document each mass resolution check by printing the MassLynx "Experiment Calibration Report," which provides a peak profile includes the result of the peak width measurement (performed at 5% of the maximum, which corresponds to the 10% valley definition. This report also allows for the determination of the resolution.

12.2 Data Acquisition

12.2.1 The total cycle time for data acquisition must be <1 second. The total cycle time includes the sum of all the dwell times and voltage reset times.

12.2.2 Acquire SIM data for all the ions listed in the five descriptors (TABLE 10).

12.3 Window Define / GC Column Performance- Window Define / GC Column Performance must be demonstrated (and documented) to be acceptable before further analysis can be performed, including initial calibrations.

12.3.1 Inject 1 µL of the Window Define Mixture (WDM; Section 8.4) and acquire selected ion monitoring (SIM) data as described above (Section 12.2).

12.3.2 The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other unlabeled TCDD isomers must be resolved with a valley of at least 25%. This is verified using the following equation:

$$\text{Valley Percent} = \frac{X}{Y} \times 100$$

Where:

X = Trough height measured between 2,3,7,8-TCDD and its closest eluting isomer

Y = Peak height of 2,3,7,8-TCDD

12.3.3 The WDM also contains the known first and last PCDD/PCDF eluters under the conditions described in Section 12.1. The retention times of these isomers are used to determine the five homologue retention time windows that are used for qualitative (Section 13.5.1) and quantitative purposes (Section 14.2.1). All peaks, including $^{13}C_{12}$-2,3,7,8-TCDD, are labeled and identified on the chromatograms. Individual selected ion current profiles (SICPs) are presented for each homologous series, as well as labeled compounds

12.3.4 Retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous is indicated in the SICP. Accurate switching at the appropriate times is necessary for accurate monitoring PCDDs/PCDFs. Particular care must be exercised for the switching time between the last tetrachlorinated congener (1,2,8,9-TCDD) and the first pentachlorinated congener (1,3,4,6,8-PeCDF), which elute within 15 seconds of each other on the 60-m DB-5 column. Corrective action is required if these congeners cannot be resolved.

12.4 Initial Calibration - Initial calibration (ICAL) is required before any samples may be analyzed for PCDDs and PCDFs. Initial calibrations are performed annually and after significant instrument maintenance such that an acceptable continuing calibration

cannot be obtained following stabilization. .

12.4.1 Tune the instrument according to section 12.1.1.2.

12.4.2 Inject 1µL of the GC Window Define solution and acquire SIM mass spectral data. Verify that the criteria for this column performance check (Section 13.2.2) is met before proceeding.

12.4.3 Using the same GC and MS conditions, analyze a 1µL portion of each calibration solution in order of lowest to highest concentration. Each point in the calibration must meet the conditions for ion abundance ratio and signal-to-noise ratio outlined in section 12.4.7. If these criteria are not met, identify the cause of the error, perform appropriate corrective action and reanalyze the entire ICAL.

12.4.4 Calculate the 17 relative response factors (RF) for the target analytes [RF_n; n = 1-17, TABLE 4]. The response factors are relative to the target analytes and the RFs for the internal standards [RF_{is}; is = 18 to 32]. The response factors for the cleanup standard [RF_{cs}, cs = 33] is also related to the recovery standard using the following equations:

$$RF_n = \frac{(A_n^1 + A_n^2) \times Q_{is}}{(A_{is}^1 + A_{is}^2) \times Q_n} \qquad RF_n = \frac{(A_{is}^1 + A_{is}^2) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{is}}$$

$$RF_n = \frac{(A_{cs}) \times Q_{rs}}{(A_{rs}^1 + A_{rs}^2) \times Q_{cs}}$$

Where:

A_n^1 and A_n^2 = sum of the integrated ion abundances of the quantitation ions for unlabeled PCDDs/PCDFs.

A_{is}^1 and A_{is}^2 = sum of the integrated ion abundances of the quantitation ions for the internal standard PCDDs/PCDFs.

A_{rs}^1 and A_{rs}^2 = sum of the integrated ion abundances of the quantitation ions for the labeled recovery standards.

A_{cs} = integrated ion abundance of the quantitation ion for the labeled cleanup standard.

Q_{is} = quantity of the internal standard injected (pg).

Q_{rs} = quantity of the recovery standard injected (pg).

Q_{cs} = quantity of the clean up standard injected (pg).

Q_n = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

12.4.5 Calculate the mean response factors for the five calibration solutions:

$$\overline{RF_n} = \frac{\sum_{j=1}^{x} RF_{n(j)}}{x} \qquad \overline{RF_m} = \frac{\sum_{j=1}^{x} RF_{m(j)}}{x}$$

Where

n = The particular native congener (n = 1 to 17)

m = The particular labeled congener (m = 18 to 26)

x = The number of calibration injections, or solutions

j = The injection number (or calibration solution number)
RF_n = Relative response factor of a particular native compound (*n*) relative to an appropriate internal standard, as determined from each injection
$\overline{RF_n}$ = Calculated mean relative response factor of a particular native compound (*n*) relative to an appropriate internal standard, as determined from the initial calibration injections (*j*)
RF_m = Relative response factor of a particular internal standard (*m*) relative to an appropriate recovery standard, as determined from each injection
$\overline{RF_m}$ = Calculated mean relative response factor of a particular labeled standard (*m*) relative to an appropriate recovery standard, as determined from the initial calibration injections (*j*)

12.4.6 The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

12.4.6.1 For congeners that belong to a homologous series with only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, and TCDF), the mean RF used will be the same as the mean RF for that single isomer.

12.4.6.2 For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer, the mean RF used for those homologous series is the mean of the RFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RF_k} = \frac{1}{t}\sum_{n=1}^{t} RF_n$$

Where:
k = 34 to 37; with 34=PeCDF; 35=HxCDF; 36=HxCDD; and 37=HpCDF (TABLE 4)
t = total number of 2,3,7,8-substituted isomers present in the calibration solutions for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

12.4.7 Criteria for Acceptable Calibration – The criteria listed below for acceptable calibration must be met before sample analyses are performed.

12.4.7.1 The percent relative standard deviations for the mean response factors [$\overline{RF_n}$ and $\overline{RF_m}$] for each native and labeled isomer must meet the criteria outlined in TABLE 6, for the appropriate method.

12.4.7.2 The Ion Abundance Ratio for all native and labeled isomers must be the criteria outlined in TABLE 9.

12.4.7.3 For each selected ion current profile (SICP) and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10.0.

12.4.8 Secondary Source Verification – Immediately following the analysis of the five calibration solutions, analyze the *Initial Calibration Verification Standard* (Section 8.6) to verify the accuracy of the calibration. The quantified values of this analysis must be within the criteria listed in TABLE 6.

12.4.9 Initial Calibrations are required annually, or under the following circumstances:

12.4.9.1 When a CCAL does not yield acceptable results after appropriate corrective actions;

12.4.9.2 Following significant instrument maintenance where subsequent column performance/continuing calibration verifications do not meet criteria once stabilized.

12.5 Instrument Sequence:

12.5.1 Once an acceptable initial calibration has been performed and verified through the analysis of the second source verification standard, sample analysis may commence. The following is an example of a typical continuous analytical sequence:

1A Window Define Mix/Column Performance Check
2A CS3
3A Method or Solvent Blank
4A Batch QC/Samples
1B Window Define Mix/Column Performance Check
2B CS3
3B Method or Solvent Blank
4B Batch QC/Samples
1C .....

12.5.2 In the sequence above items 1A through 4A represent the first 12-hour run sequence immediately followed by the next. The key requirements for each sequence are as follows:

12.5.2.1 A Mass Resolution Check is required before and after each 12 hour sequence, and must meet criteria outlined in Section 13.2.1.

12.5.2.2 The time of injection of the last sample must not be greater than 12 hours after the injection of the Window Define Mix/Column Performance Check.

12.5.2.3 Instrument conditions must remain constant during the 12-hour sequence, and no injections may occur between the last injection of the 12 hour period and the ending mass resolution check.

12.5.2.4 For Method 8290A, and some specified contract requirements, a CS3 is required to be analyzed successfully at the end of the 12-hour sequence. In this case, the time of injection of the CS3 must not be greater than 12 hours after the injection of the Window Define Mix/Column Performance Check. This verification check must meet the criteria outlined in TABLE 6.

12.5.2.5 When an ending CS3 meets the criteria for an opening CS3, it can serve as the opening CS3 for the following sequence (1B). In this case, the start of the 12-hour shift is the injection time of the CS3.

12.5.2.6 A System Blank *or* Method Blank is at the beginning of the sequence.

12.6 Extract Analysis:

Allow the extract to reach near dryness, add 20µL of recovery standard (Table 3) to the final dry extract. Mix the extract well using the vortex

12.6.1 Inject a 1µL aliquot of the extract into the GC, and analyze via the DB-5 column.

12.6.2 Acquire SIM data - Use the same acquisition and mass spectrometer operating